Application Note – Automated Switching between Fuel Cell Operation and Fuel Crossover / ECSA Diagnostics Mode with the 850 Fuel Cell Test Station

Introduction

This application note describes use of the 850 Fuel Cell Test System and accessories to perform completely automated transition from normal PEM fuel cell operation to *in-situ* electrochemical fuel crossover and electrochemical surface area measurement. The hardware and software components required are described and example data are presented.

Background – Why Perform Fuel Crossover and ECSA Measurements?

Fuel crossover rate, whether H_2, methanol or other fuel, is an important property of PEM fuel cells and is often used as an indicator of membrane health and cell failure. In addition to the fuel crossover rate, this *in-situ* method can also reveal the presence and magnitude of electrical short within the cell. The fuel crossover rate is typically measured after cell assembly (*i.e.*, beginning-of-life) as one measure of the cell build quality. A crossover rate threshold is also frequently used as an end-of-life criteria, for example in durability or accelerated stress testing to define cell failure.

Electrochemical surface area (ECSA) and catalyst utilization are also commonly determined properties of PEM fuel cells. ECSA is determined using conditions similar to those used for crossover rate determination and therefore are commonly performed back-to-back. ECSA is a measure of the active area of the catalyst of the fuel cell and is indicative of the quality or effectiveness of the electrode production or application method and materials. Utilization is determined from the ECSA and quantifies the proportion of the catalyst available to participate in the electrochemical reactions *vs*. the total area of catalyst in the electrode layer. *In-situ* ECSA measurement is a common method employed in electrode materials development (catalyst, catalyst support and ionomer), catalyst layer (electrode) manufacturing method development, and electrode degradation studies.

The principle and features of the *in-situ* fuel crossover, ECSA and voltammetry are described in the references cited at the end of this application note. It is recommended that users be familiar with these techniques before attempting to implement the fully-automated method described below.

System Requirements

1. 850C or 850e Fuel Cell Test System
2. 885 Fuel Cell Potentiostat
3. 850 Auto Multigas System
4. *FuelCell* (ver 4.0e) or later with auxiliary fuel control capability “fuelaux.ini” installed
5. Anode Reactants: Hydrogen (or methanol or other fuel)
6. Cathode Reactants: Air or oxygen for normal fuel cell operation, nitrogen for *in-situ* crossover / ECSA measurement
7. PEM (or DMFC) fuel cell

Setup

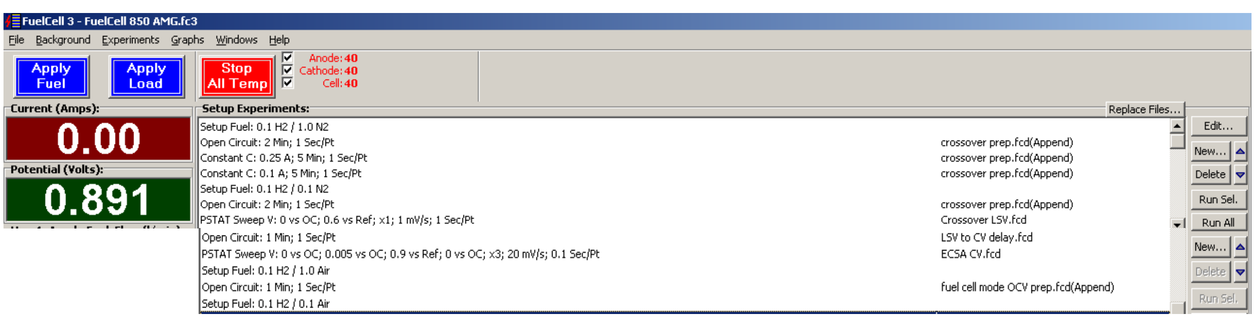
1. Install the 850 Fuel Cell Test System
2. Install the 885 Fuel Cell Potentiostat
3. Install the 850 Auto MultiGas (AMG). The following gases are required and should be plumbed to the indicated inlets on the 850 AMG:
    a. H_2: Anode Inlet A
    b. Air (or oxygen): Cathode Inlet A
    c. N_2: Cathode Inlet B
    d. Note: It is recommended that unused inlet ports on the 850 AMG be capped
4. Install *FuelCell* software version 4.0e or higher and the fuelaux.ini file as described in the AMG installation and setup guide.
5. Download the *FuelCell* setup file "850-AMG.fc3" from www.scribner.com/files/amg/850-AMG.fc3 or contact Scribner Associates at support@scribner.com.
    a. This *FuelCell* setup file consists of an experiment list that will execute the transition of the fuel cell from normal operating mode to the crossover / ECSA operating mode, perform the LSV and CV scans.
    b. Notes: Parameters of the experiments in this lets should be changed as needed. For example, the flow rates, cell and humidifier temperatures, duration of delays, LSV and CV scan rate, number CV cycles, *etc.* can be selected to obtain the desired test conditions.
    c. The default conditions are generally suitable for a 25 cm^2 H_2 PEM cell with diagnostics performed at 40 ^oC and 100% RH inlet gases.

General Procedure

The *FuelCell* setup file "FuelCell 850 AMG.fc3" contains the following list of experiments used automatically perform the LSV (for fuel crossover) and CV (for ECSA) using an 850, 885 Potentiostat and 850 AMG.

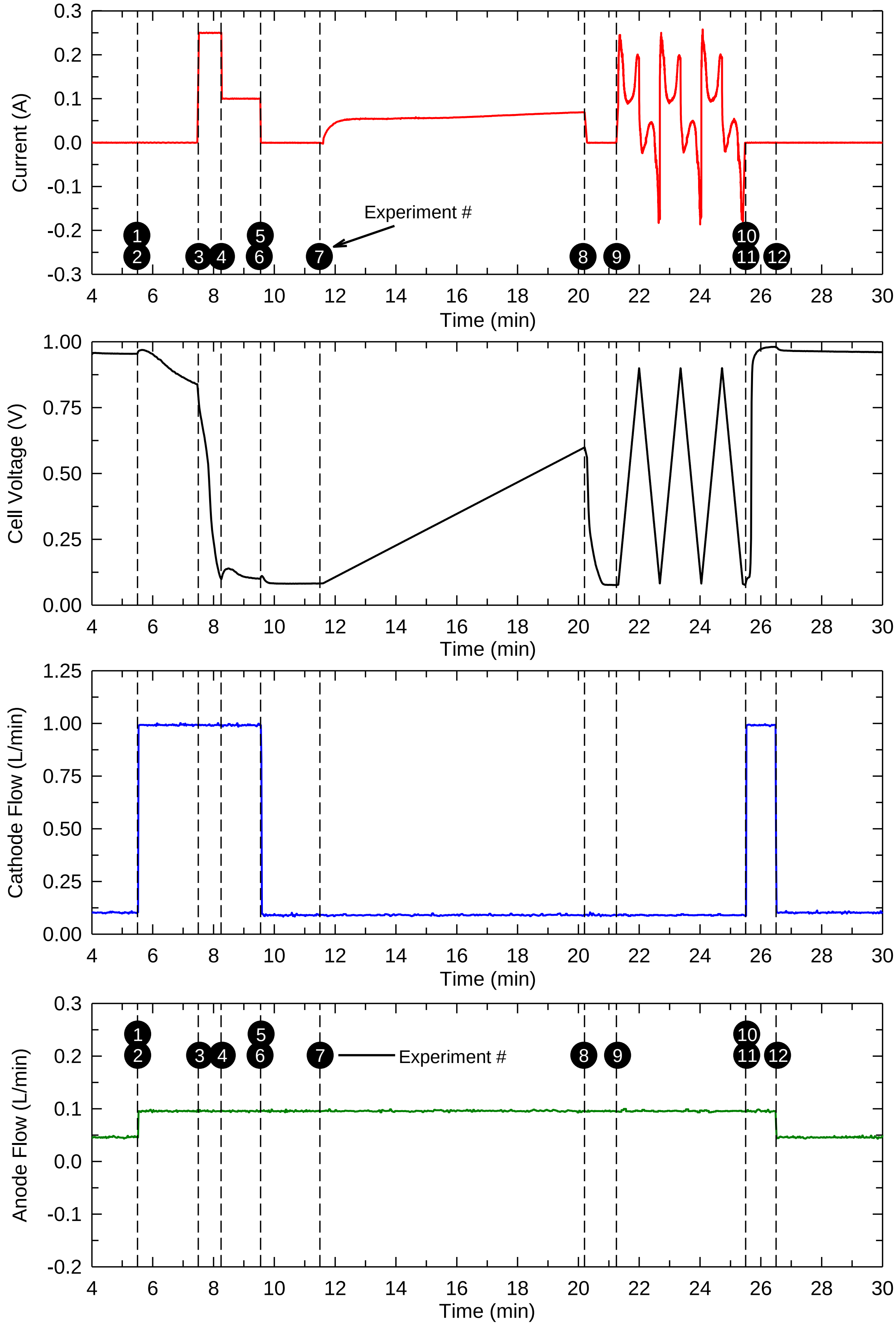
No.	Experiment Type	Default Parameters	Important Experiment Parameters	Comment
1	Setup Fuel	0.1 SLM H_2 / 1.0 SLM N_2, 40 °C humidifiers	N_2 flow rate, humidifier temperatures	Switch to N_2 on cathode; set high N_2 flow rate to purge O_2
2	Open Circuit	2 min	Duration	Delay while cathode purges with N_2
3	Constant current	0.25 A, 5 min, terminate when $E_{cell} < 0.1$ V	Current, duration, experiment terminate voltage	Apply small current to consume residual O_2 to decrease cell OCV
4	Constant current	0.1 A, 5 min, terminate when $E_{cell} < 0.1$ V	Current, duration, experiment terminate voltage	Apply small current to consume residual O_2 to decrease cell OCV
5	Setup Fuel	0.1 SLM H_2 / 0.1 SLM N_2, 40 °C humidifiers	Flow rates, humidifier temperatures	Decrease N_2 flow rate to value appropriate for crossover / ECSA
6	Open Circuit	2 min	Duration	Delay while cell voltage stabilizes; should be < 0.1 V
7	PSTAT Sweep	Voltage scan from 0 V *vs.* OCV to 0.6 V *vs.* REF at 1 mV/sec	Sweep rate, final potential	LSV for crossover and electrical short
8	Open Circuit	1 min	Duration	Delay while cell voltage stabilizes after LSV; should be < 0.1 V
9	PSTAT Sweep	Voltage scan between Vertex #1 = 0.005 V *vs.* OCV and Vertex #2 = 0.9 V *vs.* REF, 20 mV/sec, 10 points/sec, 3 cycles	Sweep rate, Vertex #1 and Vertex #2 potentials, number of cycles, points/sec	3 CVs for ECSA
10	Setup Fuel	0.1 SLM H_2 / 1.0 SLM Air, 40 °C humidifiers	Flow rates, humidifier temperatures	High air flow rate to purge cathode of N_2, prepare for fuel cell operating mode
11	Open Circuit	1 min	Duration	Delay while cathode purges with airV
12	Setup Fuel	0.1 SLM H_2 / 0.1 SLM Air, 40 °C humidifiers	Flow rates, fixed or stoichiometric flow rate control, humidifier temperatures	Establish normal operating flow rates

Screenshots of the Experiment List in the *FuelCell* setup file "FuelCell 850 AMG.fc3"

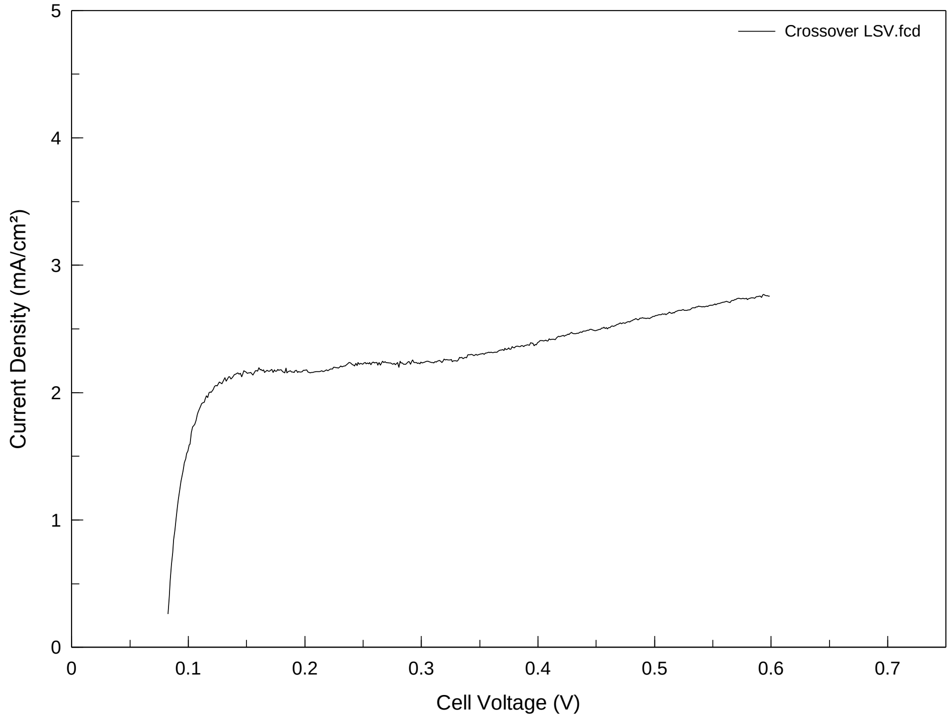


Sample Data

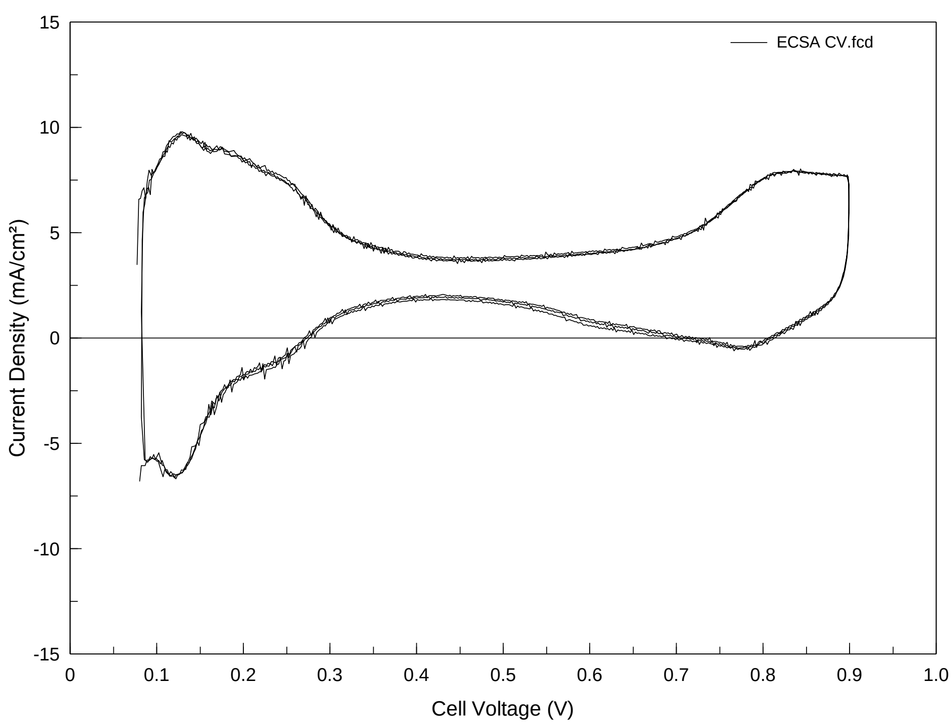
As an example of this procedure, the cell voltage, current, and anode and cathode flow rates are shown in following graphs. Individual experiments are indicated by the dashed lines.


Current (A)
Time (min)
Experiment #
1
2
3
4
5
6
7
8
9
10
11
12
Cell Voltage (V)
Cathode Flow (L/min)
Anode Flow (L/min)

Voltammogram from the LSV used for H_2 crossover rate ~ 2 mA/cm^2. The cell exhibits a significant electrical short as well, as evidenced by the positive slope at E_{cell} > 0.3 V.



Data from Cyclic Voltammetry used to determine ECSA by the hydrogen adsorption / desportion method.



Suggested Reading and References

General

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In-situ **Fuel Crossover Rate and E;ectrical short by Linear Sweep Voltammetry**

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In-situ **Electrochemical Surface Area by Cyclic Voltammetry**

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