Recommendation for Measurements of Volt-Ampere Characteristics of Fuel Cells for Diagnostic Purposes

1. Preface

The present Recommendation concerns the technology of the measurements and the requirements to the instrumentation hardware and software needed for acquisition of data, necessary for diagnostic of the State of Health (SoH) of Solid Oxides Fuel Cells. The Recommendation complies totally with the Harmonized Testing Procedures [1,2]. However, it specifies some increased requirements towards the measuring instruments and provides for detailed description of the experimental technology.

The Volt-Ampere (V-A) Characteristics (VAC) are the main fundamental and the simplest description of cells performance. The deviation of the VAC out of preliminary defined limits can serve successfully for immediate and early signal for cell degradation (EWOS – Early Warning Output Signal). The periodic measurement of VACs during testing of cells and stacks life can serve for determination of those limits. The correspondent diagnostic procedure is based on the analysis of the Differential Resistance of the object.

As far as every differentiation increases the data noises, the diagnostic success requires acquisition of more precise and confident data.

2. Basic Principle

The basic principle for measurement of the Volt-Ampere characteristics is described in details in [2]. The approach is previewing a step-wise increase of the current up to a given limit and a step-wise decrease back to zero current. Thus a direction (up or down) is given for the VAC measurement which consists of a number of steps, i.e. mini-cycles.

Every mini-cycle has a selected duration (hold time) and contains two periods - Delay time and Data Acquisition time. The delay time is necessary (un-avoidable) for attaining a stable steady state of the object.

After every change of the current to a new fixed value starts a relaxation process of the voltage. A variety of different processes with different speed (Time-constants) is taking place at the anode, at the cathode and in the other components of the cell. Their time-constants are different and depend on the natural properties of the cell (stack) determined by it architecture, technology, external conditioning, state of the object (system) etc. When the state of the object relaxes, a stationary state is achieved.

3. Delay Time and Acquisition Time

In order to provide for precise measurements, a delay time before the measurement is necessary. The delay time is the period of time necessary for full relaxation to attain stationary state of the object for the new value of the current under the selected external conditioning.

After reaching the stationary state (after the end of the delay time) the acquisition time is starting. This is the period of time, during which the measurements of valid stationary data are performed. The simplest way is a measurement of a single vector of data (time, voltage and current), however measurement of a set of data during the acquisition time is recommended. They provide for enhanced selectivity, precision and post-experimental assessment of the data stationary state and confidence.

4. Full Algorithm

The full algorithm for a precise VAC measurement comprises several steps:

- Preconditioning of the object: after reaching the desired temperature, the external gas supply conditioning should be also performed. The required gas flows (fuel and oxidant) depend on the cell size and technology and on the test type.

  In case of pristine cells it is highly recommended to perform a preliminary operating of the cell at a current set to attain a voltage of 0.85 V per cell. The time of this preparatory operation should not be less than 1 hour.

- OCV measurement: in this step the current should be zero (really open circuitry) and after delay time of 10 minutes the continuous measurements of the cell voltage should be performed (f.e. every 1 min.) until the un-stability of the measured OCV drops to less than 0.25 mV/min during 4 minutes (or 1 mV per 4 minutes). Then a selected number of OCV measurements should be performed (f.e. 5).
- Current sweep: the galvanostatic current sweep should be performed step-wise. The value of the step should be selected in order to produce 20 to 40 points up to the expected maximal current (i.e. 0.05 A/cm^2 for 1 A/cm^2 range).

  After every step of current increase a delay time should be hold. The current and voltage measurement should continue during the delay time with selected sampling rate (f.e. every 5 seconds). These data are supplementary and can be used to check the establishment of the necessary stable state and adjustment of the delay time, as well as for diagnostic purposes during the post-experimental analysis. After the delay time the next period of the mini-cycle can be started.

  During the Acquisition time a number of voltage and current measurements are performed with the selected sampling rate. The average values of the acquired data are the registered values for this step (mini-cycle) of the VAC measurement. Then the next step of the current step-wise sweep can be started.

  The maximal value of the current sweep should be limited by one of the following criteria:

  - P_{max} – reaching the point of P_{max} (or one – two current steps higher);
  - I_{max} - given by the producer of the cell (if it exists);
  - U_{min} - recommended by the producer (or selected as 0.6 V per cell).

  The measurement of the back sweep VAC can be performed (if necessary). It follows the same procedure for every current step.

- Final OCV measurement: the VAC measurements should be finished with OCV measurement preceded by the necessary delay time.

5. Data structure

This recommendation concerns the measurement of VAC for diagnostic purposes. Taking into account this target, we can recommend the production of data files of two types – a short Functional File (FF) containing only the data of the V-A functional dependence, recalculated to single cell and cm^2 and a long Procedure File (PF) containing all measured raw data – from initial preparation till the end OCV, including the delay time data and all the measurements during the acquisition time.

In both formats, every measured point is a vector, containing time, voltage and current. In PF format this vector is expanded with the number of the mini-cycles (current step) and the number of the points inside the mini-cycle (5 parameters).

As far as the VAC is very sensitive to the temperature, as well as to the other conditioning parameters, the PF should be complemented with a Conditioning File (CF) containing those values and the time (recommended sampling rate 1 min.), or this data must be included in PF as extension of the correspondent vectors.

The PF should have a header, where the object and its preparation are included (alfa-numerical text) and a post-experimental text for observation remarks.

6. Recommended Test Parameters

The Test parameters are numerous and must be selected in accordance with the test purposes. Following the previous recommendations [1, 2] two main different test methods can be distinguished – Fast Test for fast cell qualification and Stable State method for precise measurements.

Table 1 summarises the accepted recommendations for the parameters of these test methods:

Table 1

Method	**Fast Test**	**Stable state**	**Advanced***
Current step	0.025	0.05	0.05 A/cm²
Number of steps	40	20	20**
Delay time	20	540	$dU/dt < \varepsilon_a$*** V/min
Sampling rate during delay	-	-	5 s
Acq. Time	5	60	5 s
Acq. sampling rate	1	1	1 s
V-A measurement resolution	-	-	0.005% resolution
References	[1, 2]	[2]	[this text]

Remarks

* The description of this method is given below.

** This number can vary. For research purposes the current steps and the correspondent number of points is also variable.

*** The recommended value is 0.25 mV/min.

7. Advanced VAC measurement

This method follows the Stable (steady) state method. In order to decrease the total time of the measurements and the expected errors caused by eventual instability of the external conditioning, the fixed delay time is replaced by a new parameter – the time derivative of the voltage. The total time gain is considerable, because the effective time-constant varies and can be much less in some regions of the VAC. The main target is also to increase the precision of the measurements, which is more important for the statistical-derivatives post-experimental diagnostic analysis.

The selection of the ε_a value needs a special attention. It depends on the selectivity and stability of the testing equipment. In any case this value should be 2 – 3 times higher then the natural test bench noise.

8. Requirements for the instrumentation

The successful diagnostic of the fuel cell SoH (state of health) requires the acquisition of the maximal information. To meet this target the precision of the measurements should be as high as possible. Taking into account the extremely high price of the SOFC life testing and the possibilities of the present day's technology, some recommendations can be set up:

- precision of V-A measurements	< 0.1% (0.2)%
- resolution	> 0.005 %
- sampling rates	1 s./ 5 s.
- number of current steps	20 to 40
- delay time	8 minutes
- acquisition time	2 minutes
- temperature conditioning	<= 1 ^{o}C

References

1. SOFC Single Cell Performance and Endurance Test Modules, JRC Scientific and Technical Reports, http://www.durablepower.eu/files/SOFC_SC_Performance_Endurance.pdf
2. Testing the voltage and power as function of current density Polarisation curve for a SOFC single cell, JRC Scientific and Technical Reports, http://www.durablepower.eu/files/SOFC_SC_Performance_Endurance.pdf

Proposed by: Z. Stoynov, D. Vladikova, B. Burdin (IEES-BAS)

July 2017