Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU GRANT AGREEMENT N° 621245*

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***Project Coordinator**: M. Lang – DLR*

Test Module 08: Reactant Gas Composition

Main Authors	Frederik Nygaard(DTU), Sune Veltzé (DTU)
Co-Author(s)	Xiufu Sun (DTU) Jens V.T. Høgh (DTU) Carlos Boigues (ENEA) Karine Couturier (CEA) Qingxi Fu(EIFER)

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Content

Abbreviations

ASR	Area specific resistance
nlpm	Normal litre per minute
OCV	Open circuit voltage
RU	Repeating unit
SOC	Solid oxide cell
SOFC	Solid oxide fuel cell
SOEC	Solid oxide electrolysis cell
slpm	Standard litre per minute
TIP	Test input parameter
TM	Test module
TOP	Test output parameter

TM 08 – Reactant Gas Composition

1 Objective and Scope

In this test module, the effect of the reactant gas composition on the performance of a solid oxide cell or stack is determined, relevant for both fuel cell and electrolysis operations. More specifically the test module can be used to investigate how changes in reactant gas compositions, such as inlet fuel-to-steam ratio or concentrations of impurities (e.g. sulfur compounds or other sources of catalyst poisoning impurities), affect the performance of the cells or stack, and their sensitivity.

Possible negative-electrode gases in fuel cell mode could be H_2, H_2/N_2, CH_4/H_2O, simulated reformate gases derived from natural gas or diesel using different reforming processes or other fuel mixtures such as biogas and syngas. In electrolysis mode reactant/negative-electrode gas compositions could be H_2O/H_2, H_2O/CO_2 or H_2/CO_2 or more complicated mixtures involving CO or CH_4.

To investigate performance under gas compositions where the quality of the fuel is relevant, impurities can be added separately, such as hydrogen sulfide in a hydrogen source, or similar for other sources of poisoning.

On the positive-electrode, the influence of oxygen concentration on the stack performance can be examined; also for some types of electrodes, sensitivity to steam content could be investigated. An example of objectives can be found in Table 1 , where also key parameters and short/long term effect are described.

Table 1: Possible objectives for gas variations, key parameters and long-/short term effect on the cell or stack. Recommended electrochemical evaluation methods are noted (EIS or j-V), where the j-V curve is optional in case of a constant current polarization.

Objectives	Description	Key Parameters	Noticeable Effect
Coking/carbon deposition	Carbon/steam ratio (specifically C:H:O ratios). SOFC; Effect of steam concentration on fuel electrode. SOEC Co-electrolysis.	Inlet steam and carbon containing gas on fuel electrode; $x_{H_2O,negative}$, reactant conversion (in SOEC mode co-electrolysis)	On performance & long term operation: EIS + (*j-V*)
Poisoning by reactant gas impurities	Performance loss due to impurities in reactant gases such as H_2S, e.g. adsorption on TPB.	Inlet impurities: type and concentration	On performance & long term operation: EIS + (*j-V*)
Oxygen diffusion	Sensitivity investigation of different oxygen concentrations on SOFC oxygen electrode performance.	Oxygen partial pressure	On performance: EIS + (*j-V*)

Humidification effect on negative electrode performance	Negative electrode, steam concentration	Steam partial pressure	On performance & long term operation: EIS +(*j*-*V*)
Humidification effect on positive electrode performance	SOFC positive electrode, steam concentration.	Steam partial pressure	On performance & long term operation: EIS + (*j*-*V*)
Oxidizing/reducing conditions on negative electrode side.	SOEC; H_2 and CO concentrations	Inlet H_2 and CO contents	On performance & long term operation: EIS + (*j*-*V*)

In relation to fuel or oxidant composition, it is possible to investigate the parameter variation under different test operating conditions. For example the coking/carbon deposition effect can be studied by altering the steam content and reactant gas conversion, and repeating this under different stack temperatures. So in general there is only one test objective, but the single test operating parameters can be varied as well.

All the quantities used in TM08 are defined with their symbols and units in Section 7 of TM00 “General SOC testing guidelines”. The test object for which this TM applies is also described in Section 5 of the master document TM00.

2 Test Equipment and Setup

This part is fully detailed in Section 6 of the master document TM00. A complete test system is described with all its different subsystems around as well as the interfaces between the test object and the test system. Some figures are given showing the consequent test input and output parameters’ locations on the test object as well as their measurement method and accuracy. Finally some advice is supplied in regard to the mounting of the test object in the test system and to the quality of the test environment.

3 Test Input Parameters (TIPs)

Test input parameters (TIPs) include both static and variable parameters. The static parameters do not vary for the overall duration of the test and should be kept as steady as possible. The cell or stack can be operated at nominal conditions as specified by the manufacturer or evaluated based on parameters relevant to the intended application. The variable test input parameters may vary during the duration of the TM. The SOC assembly units are usually operated in OCV or under constant current, or at a constant gas utilization/conversion. The relevant static and variable test input parameters for the reactant gas composition TM 08 are given in Table 2 and Table 3.

Table 2: Static TIPs in TM08 as defined in TM00.

Description of quantity	Symbol	Unit often used	SI unit
Active electrode area	A	cm^2	m^2
Temperature of the oven	T_{oven}	°C	K
Electrical current through the cell/stack	I	A	A
Temperature of the pre-heater for preheating the negative electrode gas stream	$T_{PH,neg}$	°C	K
Temperature of the pre-heater for preheating the positive electrode gas stream	$T_{PH,pos}$	°C	K
Flow rate of the negative electrode gas stream at cell/stack inlet	$f_{neg,in}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
Flow rate of the positive electrode gas stream at cell/stack inlet	$f_{pos,in}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$

Table 3: Variable TIPs in TM08 as defined in TM00.

Name	Symbol	Unit often used	SI unit
Flow rate of component *i* in the negative electrode gas stream at cell/stack inlet	$f_{i,neg,in}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
Flow rate of component *i* in the positive electrode gas stream at cell/stack inlet	$f_{i,pos,in}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
Mole fraction of component *i* in the negative electrode gas stream at cell/stack inlet	$x_{i,neg,in}$	-	-
Mole fraction of component *i* in the positive electrode gas stream at cell/stack inlet	$x_{i,pos,in}$	-	-

4 Test Output Parameters (TOPs)

Table 4 below shows the list of the test outputs that are recorded during the overall duration of the TM08, mainly the cell/stack assembly unit response e.g. V_{stack}, V_{cell} and $V_{RU,i}$, T_{cell}, T_{stack}, T_{TP}, T_{BP}, $T_{neg,out}$ and $T_{pos,out}$ are significant TOPs to be measured when analyzing the stack behavior under stationary and transient thermal conditions. Moreover, these TOPs allow to check the good course of the test (mainly gas tightness keeping). $f_{neg,out}$, $f_{pos,out}$, $f_{i,neg,out}$ and $f_{i,pos,out}$ as well as $x_{i,neg,out}$, $x_{i,pos,out}$, $p_{i,neg,out}$ and $p_{i,pos,out}$ are also relevant TOPs to be followed in order to check the performance and healthiness of the stack, especially when analyzing hydrogen production in SOEC operation and gas tightness.

Table 4: Test output parameters for TM08.

Name	Symbol	Unit often used	SI unit
Voltage of repeating unit (RU) *i* in the stack	$V_{RU,i}$	V	V
Voltage of the stack	V_{stack}	V	V
Temperature of the negative electrode gas stream at cell/stack inlet	$T_{neg,in}$	°C	K
Temperature of the negative electrode gas stream at cell/stack outlet	$T_{neg,out}$	°C	K
Temperature of the positive electrode gas stream at cell/stack inlet	$T_{pos,in}$	°C	K
Temperature of the positive electrode gas stream at cell/stack outlet	$T_{pos,out}$	°C	K
Flow rate of component *i* in the negative electrode gas stream at cell/stack outlet	$f_{i,neg,out}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
Flow rate of component *i* in the positive electrode gas stream at cell/stack outlet	$f_{i,pos,out}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
Flow rate of the negative electrode gas stream at cell/stack outlet	$f_{neg,out}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
Flow rate of the positive electrode gas stream at cell/stack outlet	$f_{pos,out}$	nlpm, slpm $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
Mole fraction of component *i* in the negative electrode gas stream at cell/stack outlet	$x_{i,neg,out}$	-	-
Mole fraction of component *i* in the positive electrode gas stream at cell/stack outlet	$x_{i,pos,out}$	-	-
Partial pressure of component *i* of the negative electrode gas stream at cell/stack outlet	$p_{i,neg,out}$	mbar, kPa	$N\ m^{-2}$ (Pa)
Partial pressure of component *i* of the positive electrode gas stream at cell/stack outlet	$p_{i,pos,out}$	mbar, kPa	$N\ m^{-2}$ (Pa)

5 Derived Quantities

Table 5 lists the derived quantities, which can be calculated from TIPs and TOPs. The electrical current density *j* through the cell/stack is defined as:

$$j = \frac{I}{A} \tag{1}$$

The two gas utilizations $U_{gas,neg}$ and $U_{gas,pos}$ are calculated as follows:

Number of repeating units in the stack: N

Flow rate of reactant component i (i = 1 … n) in the negative/positive electrode of the stack: $f_{i,in}$ (nlpm)

Theoretical current (I_{theory}) assuming 100% gas utilization (all reactant gas is consumed through electrochemical reactions):

$$I_{theory} = \frac{F}{V_m \times 60} \cdot \frac{\sum_{i=1}^{n} z_i \times f_{i,in}}{N} = \frac{96485.3}{22.414 \times 60} \times \frac{\sum_{i=1}^{n} z_i \times f_{i,in}}{N} = 71.74 \times \frac{\sum_{i=1}^{n} z_i \times f_{i,in}}{N} \quad (2)$$

gas utilization at current I:

$$U_{gas} = \frac{I}{I_{theory}} \times 100\% = \frac{I \times N}{71.74 \times \sum_{i=1}^{n} z_i \times f_{i,in}} \times 100\% \quad (3)$$

The average RU voltage as:

$$V_{RU,av} = \frac{\sum_{i=1}^{N} V_{RU,i}}{N} \quad (4)$$

The area specific resistance of the cell/stack or each RU as a function of the current density:

$$ASR(j) = \left| \frac{\Delta V(j)}{\Delta j} \right| \quad (5)$$

The average stack temperature as:

$$T_{av} = \frac{T_{TP} + T_{BP} + T_{neg,in} + T_{neg,out} + T_{pos,in} + T_{pos,out}}{6} \quad (6)$$

The electrical power as:

$$P_{el} = V_{stack} \times I \quad (7)$$

(Area specific) electrical power density as:

$$P_{d,el} = \frac{P_{el}}{A \times N} \quad (8)$$

Usually for SOFC the reactant components at the negative electrode are H_2, CO, CH_4 and at the positive electrode O_2. In SOEC mode the reactant components at the negative electrode are usually H_2O and CO_2, and a sweep gas is fed to the positive electrode to extract the produced oxygen, though this is not strictly necessary. There is therefore no gas utilization at the positive electrode in SOEC mode. More detailed calculation method for the derived quantities can be found in TM00.

Table 5: Derived quantities possibly calculated during the TM08, with definition of symbols as described in TM00

Name	Symbol	Unit often used	SI unit
Electrical current density through the cell/stack	j	A m^{-2}	A m^{-2}
Gas utilization at the negative/positive electrode	$U_{gas,neg}$ and $U_{gas,pos}$	-	-
Average RU voltage of all RUs in the stack	$V_{RU,av}$	V	V
Area specific resistance	ASR	□ m^2	□ m^2
Average temperature of the stack	T_{av}	°C	K
Electrical power density	$P_{d,el}$	W cm^{-2}	J s^{-1} m^{-2}

6 Test Procedure

This specific TM is dedicated to the study of the cell/stack assembly unit's electrochemical performance or performance change when the reactant gas compositions of either the negative or positive electrodes are varied. The variation may occur at OCV or at constant current.

6.1 Critical Parameters and Parameter Controls

Special care should be asserted to measurement accuracy and fulfilling stabilization criteria, as described in TM00. It is recommended using cell/stack performance as evaluation parameter for investigating the influence of reactant composition; therefore degradation should be minimized during the reactant composition characterization. For degradation study, it is recommended to refer to TM12.

6.2 Preconditioning of the Stack

Preconditioning of the cell or stack should carefully respect the parameter ranges specified by the manufacturer. A preconditioning procedure is described in TM00, and can be used if no procedure is given by the manufacturer.

6.3 Reactant Gas Composition Test Procedure

The TM08 starts by setting the following parameters initially as constants: oven temperature (T_{oven}), total outlet pressures (atmospheric or higher) as well as gas flow rates at the negative and positive electrodes. Optionally, depending on the test conditions, the TM may be performed under constant current conditions, in

which case the current (I) is kept constant during this TM (unless a specific constant gas utilization/conversion is investigated, in which case current may be varied at given gas composition). After each reactant gas change there is a stabilization period after which V_{cell} or V_{RU_i} and V_{stack} are measured.

The relevant static and variable test input parameters for the reactant gas composition TM 08 are given in Table 2 and Table 3. An example of the test input parameters of inlet reactant gas compositions and current density as function of time are given in Figure 1.

During the test, T_{oven} is controlled and kept constant all along the TM according to the targeted initial operating T_{cell} or T_{stack}. During the test, $T_{neg,in}$ and $T_{pos,in}$ can vary as well as T_{cell} or $T_{stack.}$ $f_{neg,in}$, $f_{pos,in}$, $f_{i,neg,in}$ and $f_{i,pos,in}$ gas flows are set to the defined start composition for a required amount of time to stabilize the cell and perform electrochemical measurements, $f_{i,neg,in}$ or $f_{i,pos,in}$ are then varied so as to obtain a desired $x_{i,neg,in}$ or $x_{i,pos,in}$. $x_{i,neg,in}$ or $x_{i,pos,in}$ are varied until a certain predefined limit is reached. Each change (step) in $x_{i,neg,in}$ or $x_{i,pos,in}$, is maintained for a required amount of time for stabilization and electrochemical measurements.

Minimum cell/RU voltage (V_{min}) in SOFC mode and maximum cell/RU voltage (V_{max}) in SOEC mode must be defined to avoid any potential damage to the cell/stack during the test.

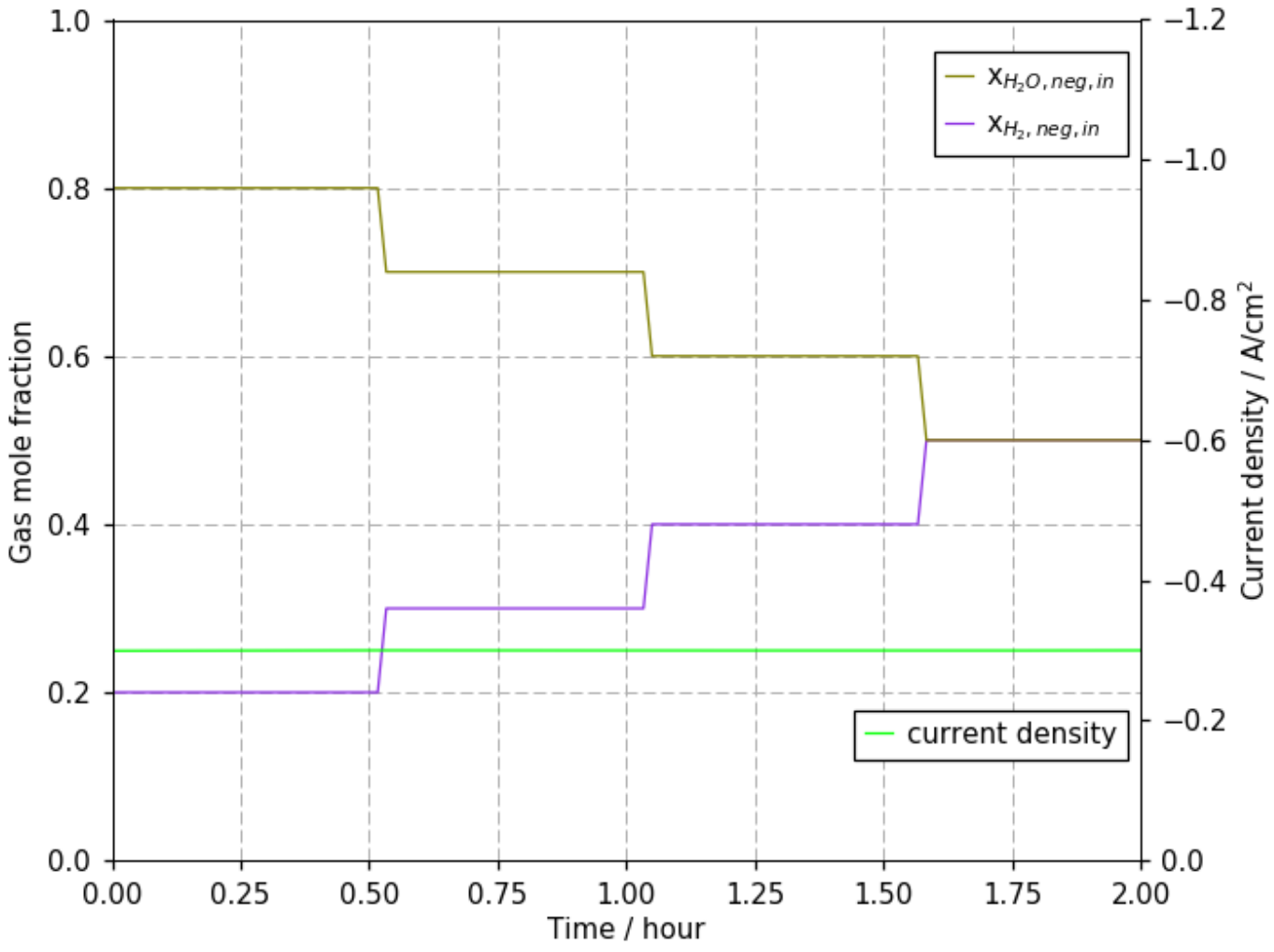

Figure 1: Example of inlet gas composition and current density as function of time in SOEC mode

7 Data Post Processing and Representation

Information on reporting of test results is mentioned in Section 9 of the master document TM00. In particular for this TM, a graph representing the evolution of RU voltages during the variation of reactant composition or current is recommended. If performed at constant current the V_{stack}, V_{cell} or $V_{RU,i}$ as a function of time / inlet gas molar fraction or catalyst poison concentration. Figure 2 shows an example of RU voltage evolution with increasing of H_2 mole fraction in SOEC operation mode at -0.3 A/cm^2.

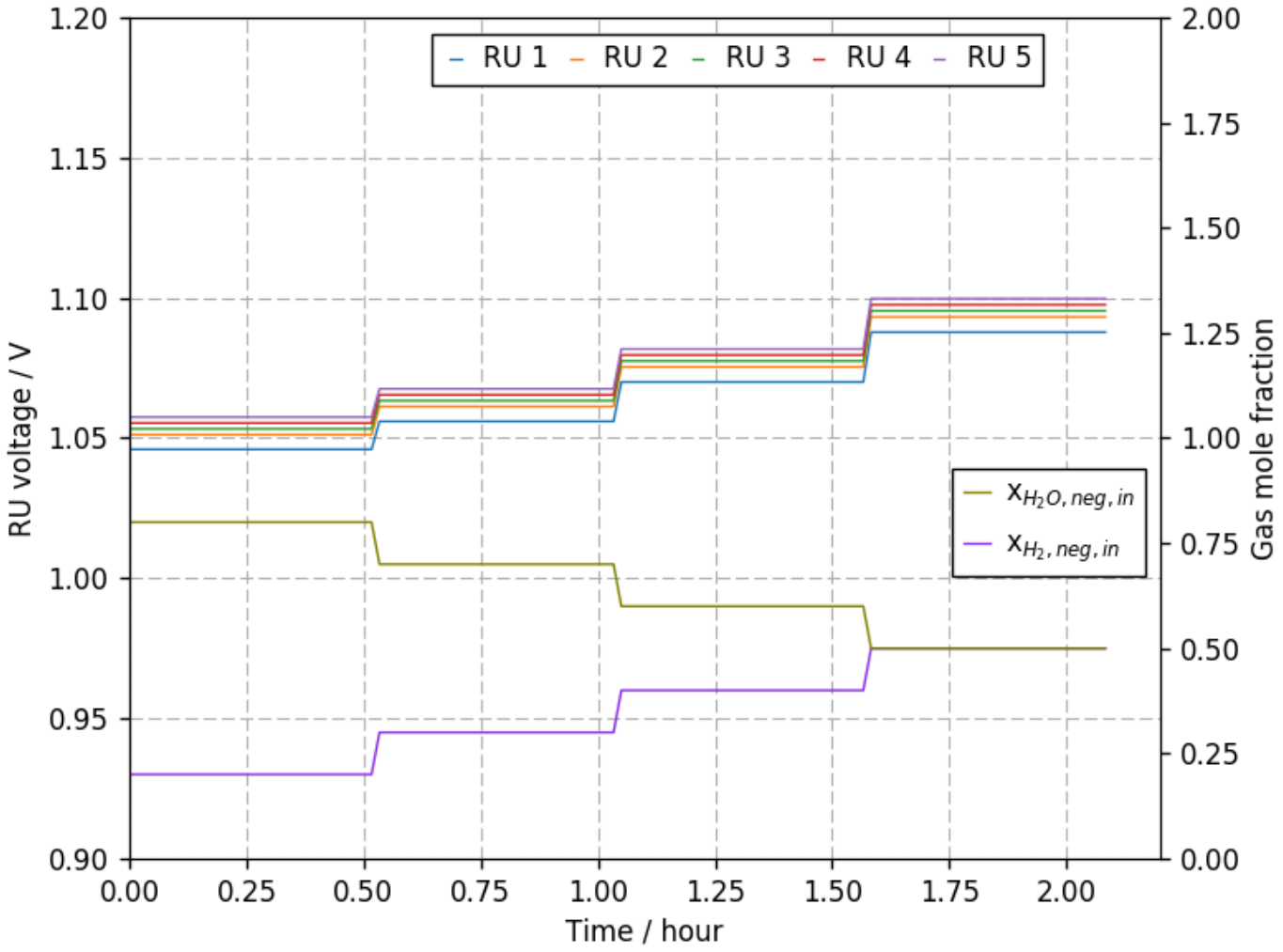

Figure 2: Example of RU voltage evolution with changing of inlet gas composition at -0.3 A/cm^2 in SOEC mode

8 Differences to Existing Procedures

The topic of this TM is quite common for performance characterization of fuel cells and can be found in many literatures e.g. [1, 2], Similar TM can be found in the STACKTEST procedures which focus on PEM-FC stacks application [2]. This TM provides more detailed test methods with test input and test output parameters which address for both SOFC and SOEC applications.

9 Bibliography

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