Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

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Test Module 02: Start-up

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Abbreviations

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 02 – Start-up

1 Objective and Scope

The objective of this test module is to define the start-up procedure needed to achieve operating conditions. Start-up includes the heating step and if needed, the tightness and electrical contact optimization, the reduction and the conditioning of the SOC cell or stack. It allows to make the SOC cell/stack operational to be tested properly on a test station.

The start-up procedure should be given by the manufacturer. If no start-up procedure is available a recommendation is given in chapter 6. In all circumstances, it is very important to distinguish between start-up of a "reduced cell/stack" and a "non-reduced cell/stack". A "reduced cell/stack" needs a reducing atmosphere at the negative electrode during start-up otherwise the nickel in the cells will re-oxidize and the cells will be destroyed. All the quantities used in TM02 are defined with their symbols and units in the section 7 of TM00 "General SOC testing guidelines". The test object for which this TM applies is also described in the section 5 of the master document TM00.

2 Test Equipment and Set-up

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)

The definition of static and variable test input parameters are defined in TM00. The relevant static and variable test input parameters for the start-up (TM02) are given in *Table 1* and *Table 2*.

The temperature change rate of the SOC cell/stack test set-up is controlled by setting the heat/cooling-rate of *the oven*, $\Delta T_{oven}/\Delta t$. The heat/cooling-rate, which can be used, is highly dependent on the test set-up, though generally 1 K min^{-1} for single cell testing [1, 3] is used.

Table 1: Static test input parameters during TM02.

Description of quantity	Symbol	Unit often used	SI Unit
Active electrode area	A	cm^2	m^2
Rate of oven temperature change	$\Delta T_{oven}/\Delta t$	°C s^{-1}	K s^{-1}

Table 2: Variable test input parameters during TM02.

Description of quantity	Symbol	Unit often used	SI Unit
Temperature of the oven	T_{oven}	°C	K
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
Pressure of the negative electrode gas stream at cell/stack outlet	$p_{neg,out}$	mbar, kPa	N m^{-2} (Pa)
Pressure of the positive electrode gas stream at cell/stack outlet	$p_{pos,out}$	mbar, kPa	N m^{-2} (Pa)
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}
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}
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)

The main output parameters of this test are the voltage of the cell, the stack and the RUs of the stack. These test outputs have to be recorded at the different test steps. The main output parameters are given in *Table 3*.

Table 3: Test output parameters for TM 02.

Description of quantity	Symbol	Unit often used	SI Unit
Voltage of the cell	V_{cell}	V	V
Voltage of the stack	V_{stack}	V	V
Voltage of repeating unit (RU) *i* in the stack	$V_{RU,i}$	V	V
Temperature of the negative electrode gas stream at	$T_{neg,in}$	°C	K

cell/stack inlet			
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
Average temperature of the stack	T_{av}	°C	K
Temperature of the stack	T_{stack}	°C	K
Temperature of the top plate of the stack	T_{TP}	°C	K
Temperature of the bottom plate of the stack	T_{BP}	°C	K
Temperature of the cell	T_{cell}	°C	K

5 Derived quantities

The following Table 4 gives the derived quantities useful for this TM. They are all calculated from TIPs and TOPs with the equations presented in TM00 - section 10.

Table 4: Derived quantities possibly calculated during TM02.

Description of quantity	Symbol	Unit often used	SI unit
Average temperature of the stack	T_{av}	°C	K
Maximum temperature difference during start-up	ΔT_{max}	°C	K

Some stack designs do not allow a direct measurement of the internal temperature of the stack. In this case an average temperature of the stack T_{av} should be calculated as a substitute for the internal temperature. The calculation can include the temperature of gases as well as the temperature of the end plates. Depending on which temperatures can be measured an average temperature can be calculated exemplary as following:

$$T_{av} = \frac{T_{TP}+T_{BP}+T_{neg,in}+T_{neg,out}+T_{pos,in}+T_{pos,out}}{6} \quad (1)$$

A stack can be damaged during the start-up if the temperature gradient between the gas inlets and the stack itself is too high. A value for the maximum temperature difference during start-up can be calculated with the following formula if the internal temperature cannot be measured directly:

$$\Delta T_{max} = \left| \frac{(T_{neg,in}+T_{pos,in})}{2} - \frac{(T_{TP}+T_{BP})}{2} \right| \quad (2)$$

6 Test Procedure

If no start-up procedure is provided by the stack manufacturer the following procedure is recommended to start-up the SOC cell/stack (see chapters 6.2 and 6.3). Recommendations for the mounting of the cell/stack is given in TM 00.

6.1 Critical parameters and parameter controls

A large temperature gradient between the gas inlets and the cell/stack itself should be avoided to reduce the risk of cell/stack damage. The rate of temperature changing $\Delta T_{oven}/\Delta t$ should not be too high in order to avoid the damage of the cell/stack.Therefore all relevant temperatures like $T_{\mathrm{cell}}/T_{stack}$, T_{TP}, T_{BP}, $T_{neg,in}$, $T_{pos,in}$, $T_{neg,out}$, $T_{pos,out}$ should be monitored precisely in order to be able to determine the maximum temperature difference during start-up ΔT_{max} (formula 2 see chapter 5). In the case if the internal temperature of the stack cannot be measured directly an average temperature of the stack T_{av} should be calculated and used (equation 1).

6.2 Start-up from cold state with non-reduced cells

1) Set the flow for the negative ($f_{\mathrm{neg,in}}$) electrode to N_2 or Ar (inert gas). Set the flow for the positive electrode ($f_{\mathrm{pos,in}}$) to air.

2) Preheating of inlet gases are initiated, $T_{PH,\mathrm{neg}}$, $T_{PH,\mathrm{pos}}$, if applicable.

3) Increase cell/stack temperature by setting T_{oven} to the sealing temperature with defined temperature changing rate $\Delta T_{oven}/\Delta t$ required by the setup. Gas inlet temperatures $T_{\mathrm{neg,in}}$ and $T_{\mathrm{pos,in}}$ are increased (if possible) by adjusting $T_{PH,\mathrm{neg}}$, $T_{PH,\mathrm{pos}}$.

4) Hold at sealing temperature according to set-up requirements.

5) Change T_{oven} to the reduction temperature required by the SOCs. Hold at the targeted value until temperature is stabilised. Change the negative electrode gas feed to H_2/N_2 (Ar) (either in a single step or step by step increase of H_2 in N_2 (Ar) content) and increase $f_{\mathrm{neg,in}}$ required for reduction. Change the positive electrode feed to air/O_2 and $f_{\mathrm{pos,in}}$ required for sintering of the electrode (if needed).

6) Hold at temperature according to the SOCs requirements.

7) Change T_{oven} to the operating temperature.

8) Change the reactant flows $f_{\mathrm{neg\ in}}$ and $f_{\mathrm{pos,in}}$ to nominal values and $x_{i,neg,in}$ to nominal composition.

9) Change the reactant pressures (both at the same time) $p_{\mathrm{neg,in}}$ and $p_{\mathrm{pos\ in}}$ (if applicable).

10) Wait until cell/stack temperature $T_{\mathrm{cell}}/T_{\mathrm{stack}}$ reaches its set-point (nominal temperature).

6.3 Start-up from cold state with reduced cells

1) Set the flow for the negative ($f_{\mathrm{neg,in}}$) electrode to a reducing flushing gas H_2/N_2 (Ar) (e.g. N_2 + 5 % H_2). Set the flow for the positive electrode ($f_{\mathrm{pos,in}}$) to air.

2) Preheating of inlet gases are initiated, $T_{PH,\mathrm{neg}}$, $T_{PH,\mathrm{pos}}$, if applicable.

3) Increase cell/stack T_{cell}/T_{stack} by setting T_{oven} to operating temperature. Gas inlet temperatures $T_{neg,in}$ and $T_{pos,in}$ are increased (if possible) to operating temperature by adjusting $T_{PH,neg}$ and $T_{PH,pos}$.

4) Hold at the targeted value until T_{cell}/T_{stack} is stabilised.

5) Change the reactant flows $f_{neg,in}$ and $f_{pos,in}$ to nominal values and $x_{i,neg,in}$ to nominal composition.

6) Change the reactant pressures (both at the same time) $p_{neg,in}$ and $p_{pos,in}$ (if applicable).

7) Wait until cell/stack temperature T_{cell}/T_{stack}, reaches its set-point (nominal temperature).

Figure 1 illustrates an example for a non-reduced SOC stack start-up procedure. During heating up of the SOC stack, N_2 is supplied to the negative electrode compartment and air is supplied to the positive electrode compartment. Stack reduction starts after the sintering of the stack with a stepwise increasing of H_2 concentration in the N_2 at the negative electrode compartment, and the increasing of air flow at the positive electrode. After reduction, the stack is ready for pre-conditioning or testing.

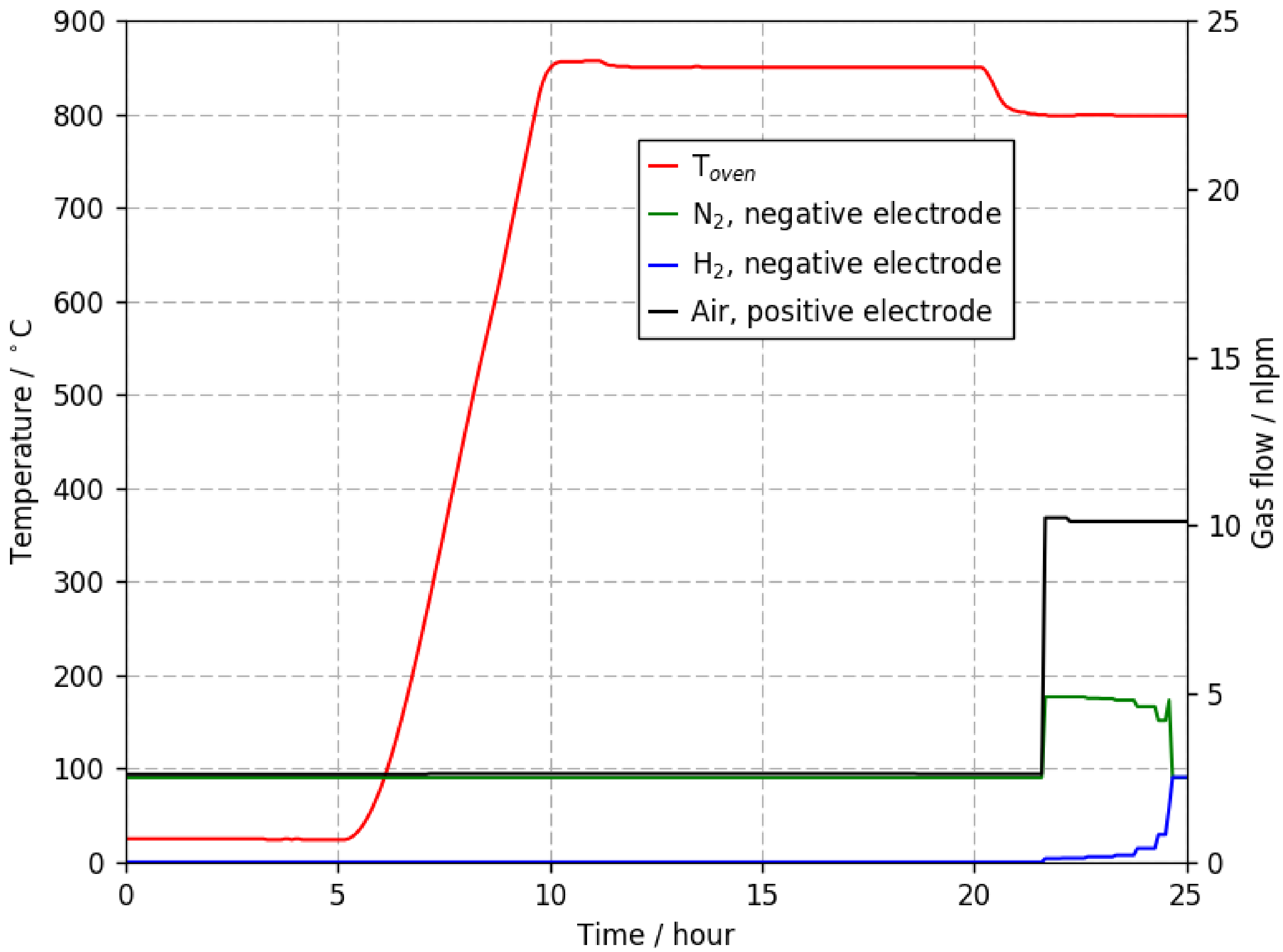

Figure 1 Schematic example of a start-up procedure for non-reduced SOC 5 cells stack

6.4 Preconditioning of the cell/stack

Preconditioning is very often needed to stabilise and increase long term cell/stack performance and it should be done according to the SOC cell or stack manufacturer. A galvanostatic pre-treatment of the cell/stack at the selected operating temperature is generally recommended to assure the electrical contact between the SOC and metallic components. The galvanostatic treatment for minimum 2 hours, at a current density of 0.3 A·cm^{-2} has been described by the FC-TESTNET [2].

7 Data Post processing and Representation

Data representation of selected TOPs can be used as an overview of the performed TM. Figure 2 shows an example of the RU voltages change during the start-up of a 5 cell SOC short stack.

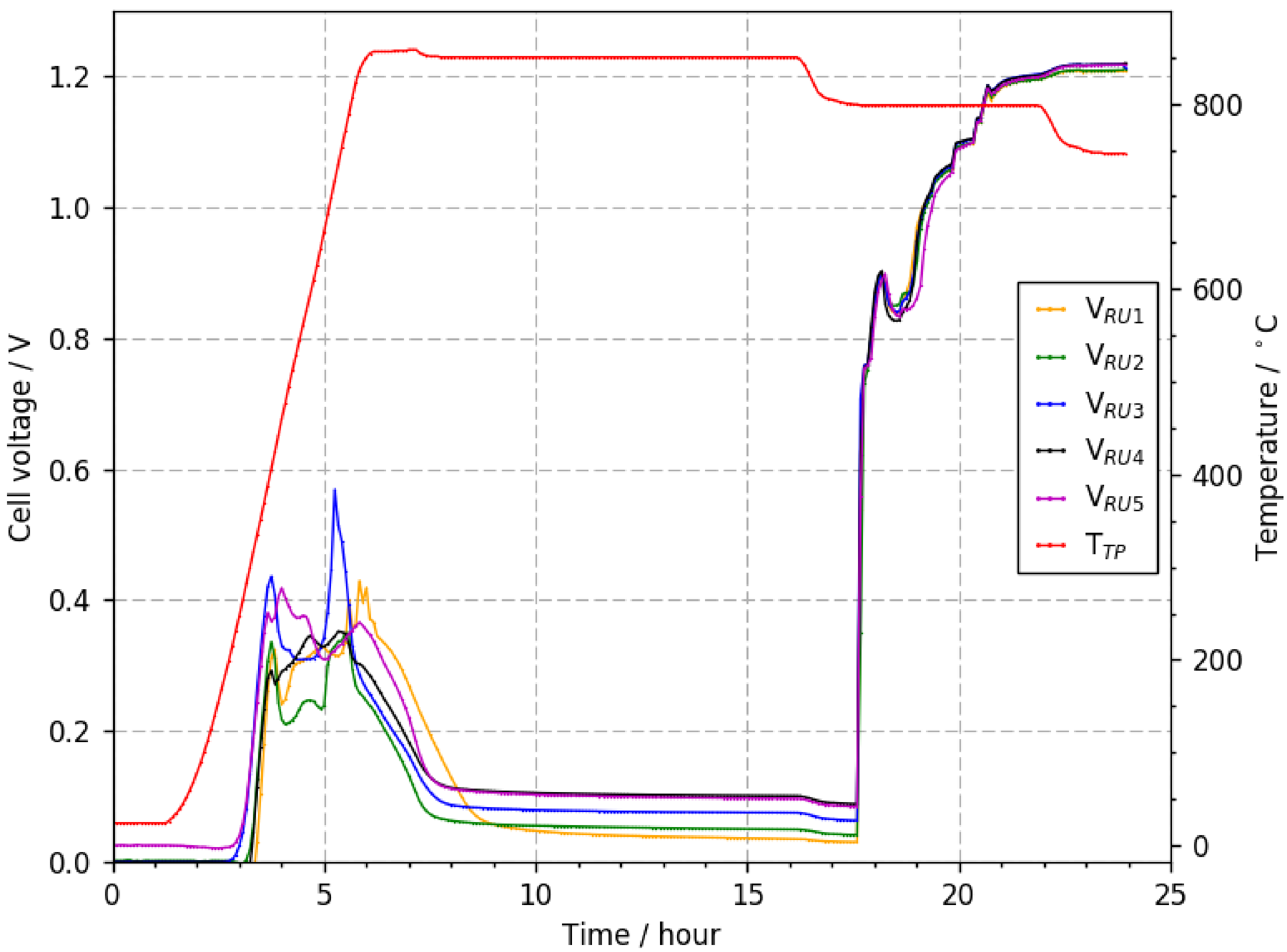

Figure 2: *Voltages and top plate temperature of the repeating units of a 5 cells non-reduced SOC short stack during start-up*

After the reduction the gas tightness of the cell/stack can be evaluated by reading of the measured RU voltages (OCV).

8 Differences to Existing Procedures

This TM topic is quite common and usually described by the cell/stack manufactures. The use of this TM also depends on the specific test facilities. Similar start-up test procedure can also be found in references [1-4]. However, the current test module gives an additional descriptive start-up sequence, which may be used for both SOC single cell and stack testing, also details the relevant TIPs, TOPs and derived quantities with their associated formularies, their evolutions.

9 Bibliography

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