



Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

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Test Module 03: Current-voltage Characteristics

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Abbreviations

ASR	Area specific resistance
HHV	Higher heating value
LHV	Lower heating value
nlpn	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 litres per minute
TIP	Test input parameter
TM	Test module
TOP	Test output parameter

TM 03 – Current-voltage Characteristics

1 Objective and Scope

This document presents the test module 03 (TM03) which deals with solid oxide cell (SOC) operation at different current densities either as a fuel cell (SOFC) or as an electrolyser (SOEC) to determine the current-voltage characteristics of a SOC cell/stack. The aim of this test module is to establish a widely accepted method for SOC performance characterisation by means of polarization (j - V) curve measurements where the voltage of the SOC is measured as a function of the current density (current-voltage characteristics). This test module addresses SOC cell/stack assembly units, testing systems, instruments, measuring methods and test methods. This test module is a general characterization method that can be used in SOC research and development and for quality assurance of SOC cell/stack. Moreover, it can be used as a baseline measurement for the qualification of an SOC in a given application. All the quantities used in TM03 are defined with their symbols and units in chapter 7 of TM00 “General SOC testing guidelines” (master document) [1]. Most importantly, the parameters, values and range of values including uncertainties used in this document are recommended only, unless otherwise noted. The test object for which this TM applies is also described in chapter 5 of TM00.

2 Test Equipment and Set-up

The test equipment is fully described in chapter 6 of the master document TM00 [1], where a complete test system is shown with all its different subsystems. Moreover, all interfaces between the test object and the test system are discussed in detail. The electrical output / input power control subsystem is described in chapter 6.3 of TM00. The j - V curves are usually measured in galvanostatic mode, which means that a defined electrical current is applied to the test object and the corresponding voltage is measured. This is usually done by connecting both current and voltage probes of the test object to an electronic load. For j - V curves the electrical current usually is increased and decreased stepwise. Different setups are necessary for testing of either cells or stacks in fuel cell (SOFC) or electrolysis (SOEC) mode. In SOFC mode for single cell and short stacks an additional voltage supply may be needed, e.g. to overcome voltage drops in the hot current wires. In SOEC mode the voltage supply is necessary in order to impose the electrolysis voltage on the SOEC cell/stack object. In this case the polarity of the cell/stack has to be reversed in the test setup compared to the SOFC mode in order to reverse the current direction. For stack measurements the current is always applied to or taken from the whole stack whereas the voltage probes can either be connected to the complete stack or are just taken from the repeat units of interest. The latter case has the advantage to examine the electrochemical behavior of individual repeat units of the stack.

3 Test Input Parameters (TIPs)

There are two types of test input parameters: variable and static (for terms and definitions refer to chapter 3 in the master document TM00). The first type may vary during the duration of the TM while the second type does not vary during the overall test duration. The SOC assembly units are usually operated in galvanostatic mode at a given operating point (static TIPs).

The TIPs (see *Table 1* and *2* below) are either recommendations by the manufacturer or are defined considering the application and objective of the test.

Table 1: Static test input parameters during the TM03.

Description of quantity	Symbol	Unit(s) often used	SI Unit
Electrical current rate of change	$\Delta I / \Delta t$	A s ⁻¹	A s ⁻¹
Flow rate of component <i>i</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>i</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
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
Temperature of the oven	T_{oven}	°C	K
Pressure of the negative electrode gas stream at cell/stack outlet	$p_{neg,out}$	mbar, kPa	N m ⁻²
Pressure of the positive electrode gas stream at cell/stack outlet	$p_{pos,out}$	mbar, kPa	N m ⁻²
Mole fraction of component <i>i</i> in the negative electrode gas stream at cell/stack inlet	$x_{i,neg,in}$	-	-
Mole fraction of component <i>i</i> in the positive electrode gas stream at cell/stack inlet	$x_{i,pos,in}$	-	-

Table 2: Variable test input parameter during the TM03.

Description of quantity	Symbol	Unit(s) often used	SI Unit
Electrical current through the cell/stack	<i>I</i>	A	A

4 Test Output Parameters (TOPs)

Table 3 below lists the test output parameters (TOPs) that are determined in the application of this test module. The most important TOP is the SOC voltage. Other TOPs may be measured and recorded as desired by the test objective.

Table 3: Test output parameters.

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 cell/stack inlet	$T_{neg,in}$	°C	K
Temperature of the positive electrode gas stream at cell/stack inlet	$T_{pos,in}$	°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 outlet	$T_{pos,out}$	°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
Stack temperature	T_{stack}	°C	K
Temperature of the cell	T_{cell}	°C	K

5 Derived Quantities

Table 4 gives the quantities derived or calculated from TIPs and TOPs with the equations presented in chapter 10 of the master document TM00.

Table 4: Derived quantities.

Description of quantity	Symbol	Unit often used	SI unit
Electrical current density through the cell/stack	j	mA cm ⁻²	A m ⁻²
Area specific resistance	ASR	Ω cm ²	Ω m ²
Electrical power of the cell/stack	P_{el}	W	J s ⁻¹
Electrical power density (area specific)	$P_{d,el}$	W cm ⁻²	J s ⁻¹ m ⁻²
Gas utilization at the positive electrode (air utilization or oxygen utilization)	$U_{gas,pos}$	-	-
Gas utilization at the negative electrode (fuel utilization in	$U_{gas,neg}$	-	-

Description of quantity	Symbol	Unit often used	SI unit
SOFC mode, steam conversion in SOEC mode)			
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 inlet	$X_{i,pos,out}$	-	-
Flow rate of component i in the negative electrode gas stream at cell/stack outlet	$f_{i,neg,out}$	nlpm, slpm $l_n \text{ min}^{-1}$, $l_s \text{ min}^{-1}$	$\text{m}^3 \text{ s}^{-1}$
Flow rate of component i in the positive electrode gas stream at cell/stack outlet	$f_{i,pos,out}$	nlpm, slpm $l_n \text{ min}^{-1}$, $l_s \text{ min}^{-1}$	$\text{m}^3 \text{ s}^{-1}$
Pressure drop of the negative electrode gas stream over the cell/stack	Δp_{neg}	mbar, kPa	N m^{-2} (Pa)
Pressure drop of the positive electrode gas stream over the cell/stack	Δp_{pos}	mbar, kPa	N m^{-2} (Pa)
Partial pressure of component i of the negative electrode gas stream at cell/stack inlet	$p_{i,neg,in}$	mbar, kPa	N m^{-2} (Pa)
Partial pressure of component i of the positive electrode gas stream at cell/stack inlet	$p_{i,pos,in}$	mbar, kPa	N m^{-2} (Pa)
Average stack temperature	T_{av}	°C	K
Average RU voltage of all RUs in the stack	$V_{RU,av}$	V	V

The derived quantities are calculated in accordance with chapter 10 of the master document TM00. The average stack temperature is

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

The average RU voltage is

$$V_{RU,av} = \frac{\sum_{i=1}^N V_{RU,i}}{N} \quad (2)$$

with the number of repeating units in the stack, N .

The electrical power of the cell / stack is

$$P_{el} = V_{cell/stack} \times I \quad (3)$$

The electrical power density of the cell / stack is

$$P_{d,el} = \frac{P_{el}}{A \times N} \quad (4)$$

with the active electrode area, A and N , the number of repeating units.

The most common method for the determination of the ASR values is the interval method. Two specific points are selected in the current voltage curve in order to calculate the slope of the curve at a specific current density. The magnitude of the area specific resistance (ASR) at a specified current density is

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

where $\Delta V(j)$ and Δj are respectively the difference in SOC voltage at a specified current density and the difference in current density corresponding to the SOC voltage difference.

For example, the ASR at 500 mA/cm^2 is determined by the ratio of the SOC voltage measured at or nearest to a current density of 510 mA/cm^2 less the SOC voltage measured at or nearest to a current density of 490 mA/cm^2 and the difference between the two corresponding current densities.

However, in the case of fluctuations of the voltage an exact determination of the stack ASR is not possible with the interval method. An alternative method is the enlargement of the interval so that the influence of the fluctuations is minimized. This measure is only applicable in the j - V curve region with almost linearity behavior. Another method is the ASR -determination using a linear regression which is mathematically fitted to the j - V curve. The corresponding mathematical algorithms are already integrated in most of the established software programs. The ASR can then be calculated with the corresponding linear equation. This method is also only applicable for linear j - V curves and is even more susceptible to errors the more the entire characteristic curve has a non-linearity.

The last method is the determination of the ASR by a regression using a polynomial. In contrast to the linear regression, the j - V curve is fitted through a higher degree equation with the polynomial regression, usually a so-called Taylor polynomial. This enables also to describe non-linear j - V curves. The corresponding equation for describing the j - V curve usually has the form:

$$V = a + b \cdot j + c \cdot j^2 + d \cdot j^3 + e \cdot j^4 + f \cdot j^5 + \dots \quad (6)$$

The ASR can be determined by calculation of two points of the j - V curve with the polynomial equation and calculating the slope within the corresponding interval. It is recommended to use the polynomial regression method for the determination of $ASRs$ of non-linear j - V curves that have voltage instabilities.

For SOFC, the electrical efficiency of the cell / stack at the higher heating value (HHV) and the lower heating value (LHV) are respectively

$$\eta_{el,LHV} = 22.414 \times 60 \times \frac{P_{el}}{\sum_{i=1}^n LHV_i \times f_{i,neg,in}} \quad (7)$$

and

$$\eta_{el,HHV} = 22.414 \times 60 \times \frac{P_{el}}{\sum_{i=1}^n HHV_i \times f_{i,neg,in}} \quad (8)$$

For hydrogen, for example, the HHV and LHV are $285.98 \text{ kJ mol}^{-1}$ and $241.77 \text{ kJ mol}^{-1}$, respectively.

For SOEC using steam and assuming 100% current efficiency, the electrical efficiency of the stack at the higher heating value (HHV) and the lower heating value (LHV) are

$$\eta_{el,LHV,H2-production} = \frac{1.253 \times N}{V_{stack}} \quad (9)$$

and

$$\eta_{el,HHV,H2-production} = \frac{1.482 \times N}{V_{stack}} \quad (10)$$

respectively.

For both SOFC and SOEC, the utilization of reactant component i ($i=1\dots n$) in the negative/positive electrode of the stack at a specified current I is:

$$U_{gas} = \frac{I \times N}{71.74 \times \sum_{i=1}^n z_i \times f_{i,in}} \times 100\% \quad (11)$$

with z_i , the number of exchanged electrons in the electrochemical cell reaction of reactant component i and $f_{i,in}$, the volume inlet flow of reactant component i with the unit of nlpm.

6 Test Procedure

The main aim of this TM is to determine the SOC voltages (V_{cell} , V_{stack} , $V_{RU,i}$) versus the current density. The duration of the test depends on the number of measured current densities and its rate of increase/decrease as well as the specified cut-off voltage.

For SOFC, a value of 0.6 V per cell is recommended as cut-off voltage. For SOEC, 1.4 V is recommended as cut-off voltage per cell. For stack measurement the cut-off voltage is related to the worst repeating unit ($V_{RU,i}$) value.

Note: Any deviation from this procedure should be described in the test report.

6.1 Critical parameters and parameter controls

The test starts by bringing the static TIPs (see *Table 1* above) to their specified values followed by a stabilization period during which the specified TIP stability criterion (criteria) is (are) to be met. During the test, all TIPs should be measured and recorded as described in the master document TM00.

It is recommended to seek stability of T_{cell} , T_{stack} , $T_{neg,in}$, $T_{pos,in}$, $T_{neg,out}$ and $T_{pos,out}$ under OCV conditions (zero current) prior to the actual polarization (j - V) curve measurement (current-voltage characteristics) and to limit gaps between all the measured temperatures for a better control of the cell/stack temperature.

In SOEC mode, special attention should be paid to a stable supply of steam in order to limit fluctuations in the voltages. The SOC voltages should not vary by more than a specified value at open circuit voltage (OCV) conditions. A threshold value of ± 10 mV per cell is recommended as stability criterion.

During the test, all TOPs should be measured and recorded as described in the master document TM00.

6.2 Measurement of Current-Voltage Characteristics

The j - V curve measurement starts at OCV up to the current where the specified cut-off voltage (ascending j - V curve) is attained and back to OCV (descending j - V curve). The TIPs and TOPs are continuously recorded at their specified sampling rates. The recommended sampling rate is 1 Hz.

The rate of change in current density depends on the maximum test duration, the thermal mass of the SOC cell / stack and the acceptable voltage difference between the ascending and descending j - V curves. A value of 1 mA/cm^2 per second is recommended for the rate of change in current density. The measured values of SOC voltage versus the current density constitute the data points of the j - V curve.

When the step duration is greater than one second, for example, with a step in current density of 30 mA/cm^2 every 30 seconds, the values of SOC voltage last measured during each step versus the current density constitute the data points of the j - V curve.

Figure 1 to Figure 6 below present examples of TIPs and TOPs recorded during j - V curve measurements.

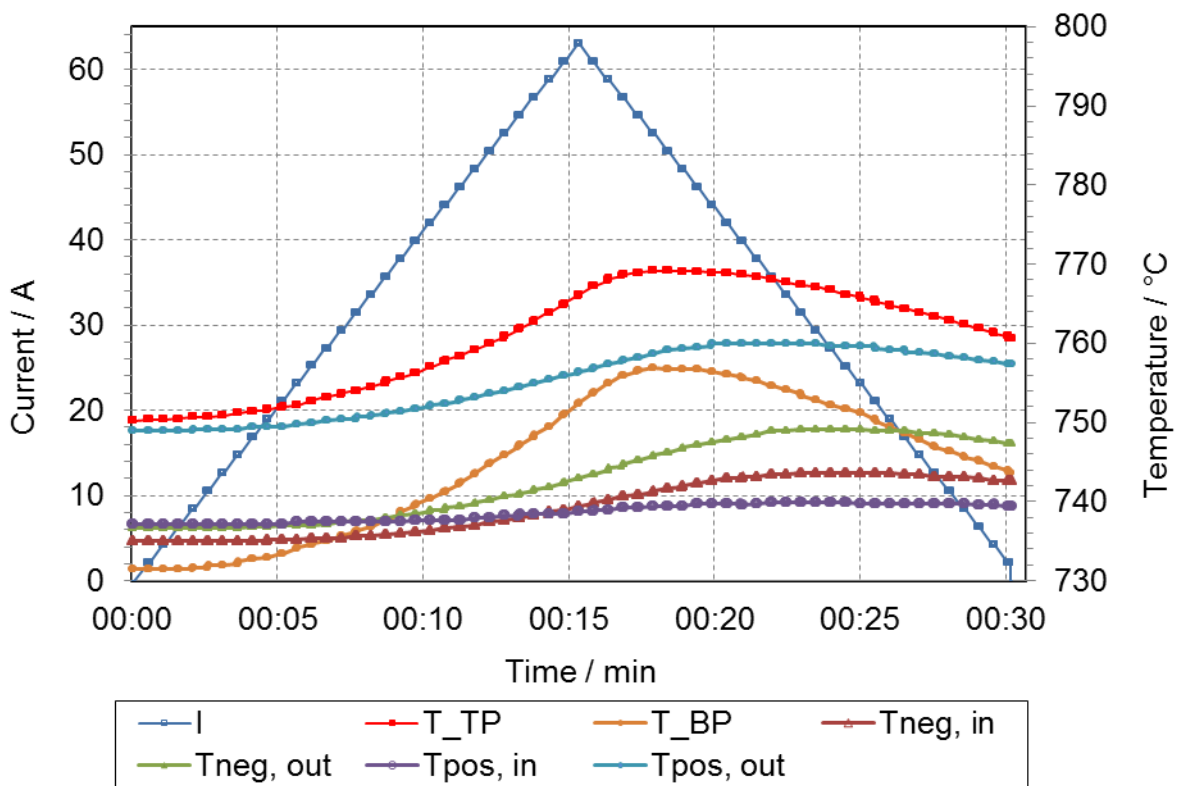


Figure 1: Example of TIP (current) and TOPs (SOFC temperatures) recorded during j - V curve measurement (ascending and descending) showing their evolution.

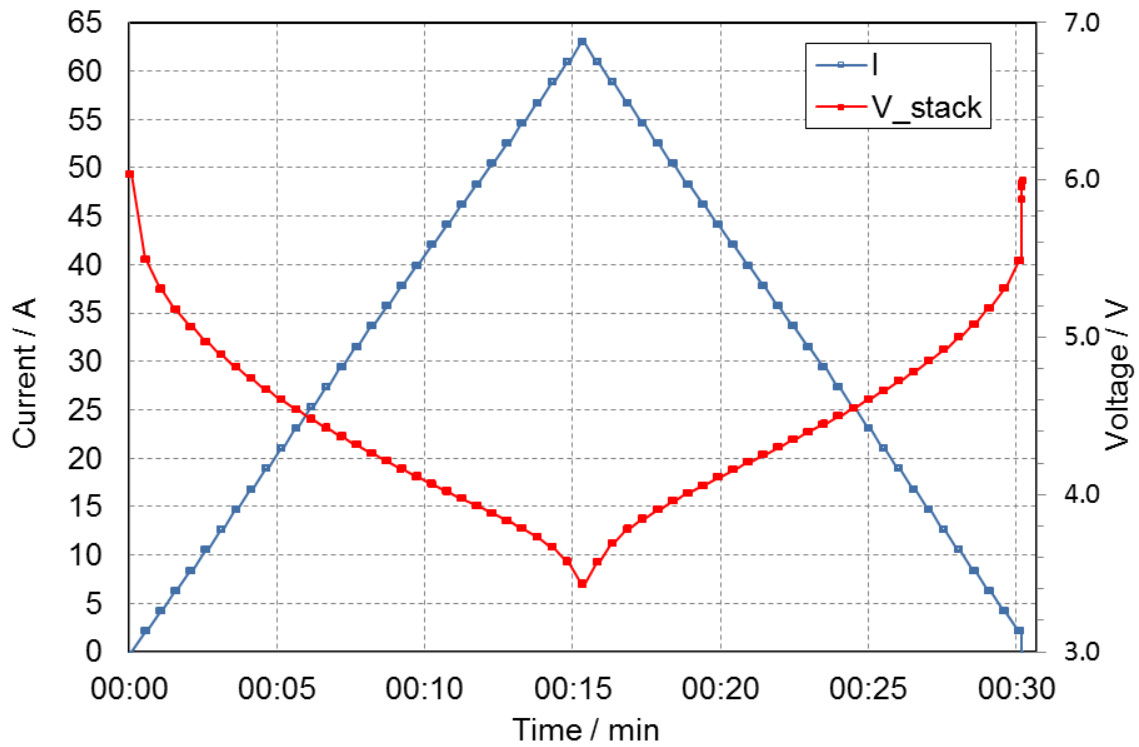


Figure 2: Example of TIP (current) and TOPs (SOFC stack voltage) recorded during j-V curve measurement (ascending and descending) showing their evolution.

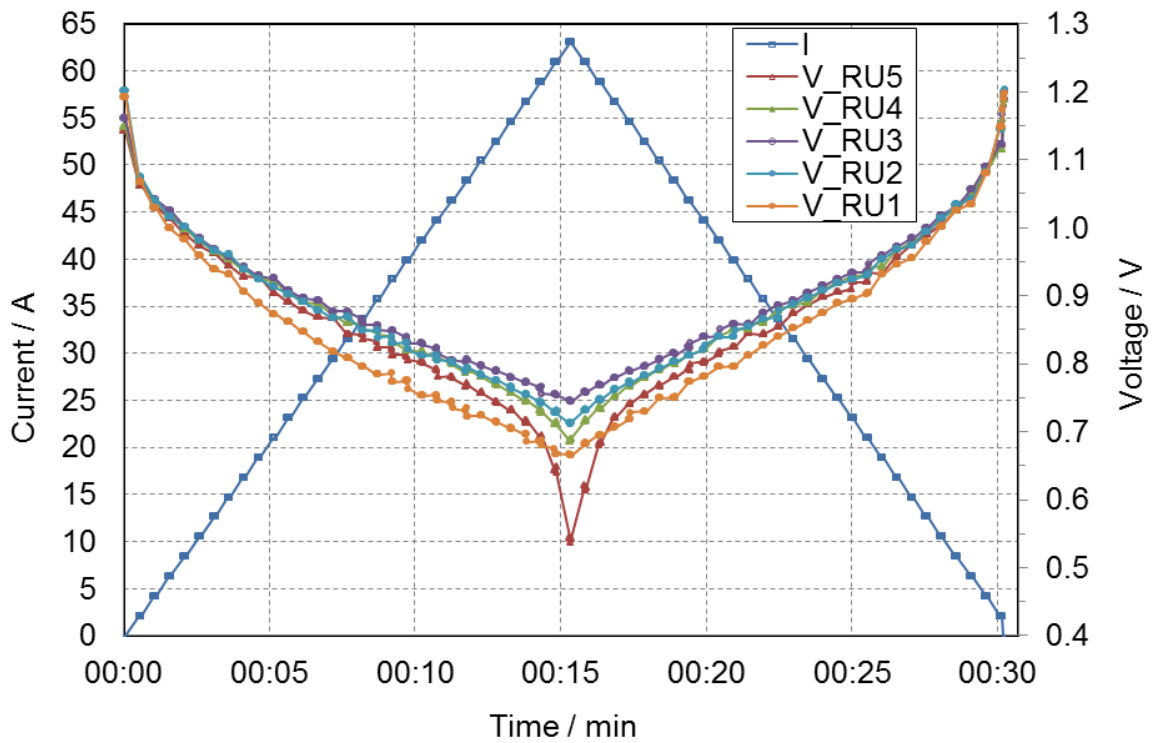


Figure 3: Example of TIP (current) and TOPs (SOFC RU voltages) recorded during j-V curve measurement (ascending and descending) showing their evolution.

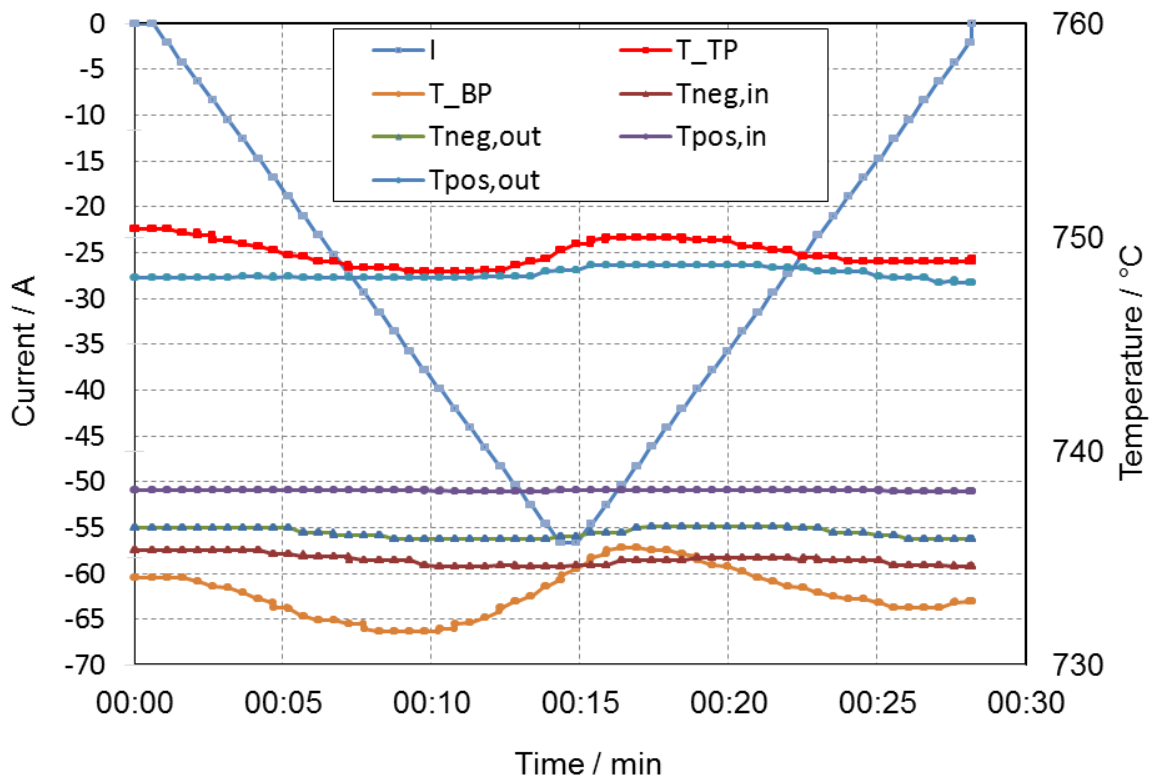


Figure 4: Example of TIP (current) and TOPs (SOEC temperatures) recorded during j-V curve measurement (ascending and descending) showing their evolution.

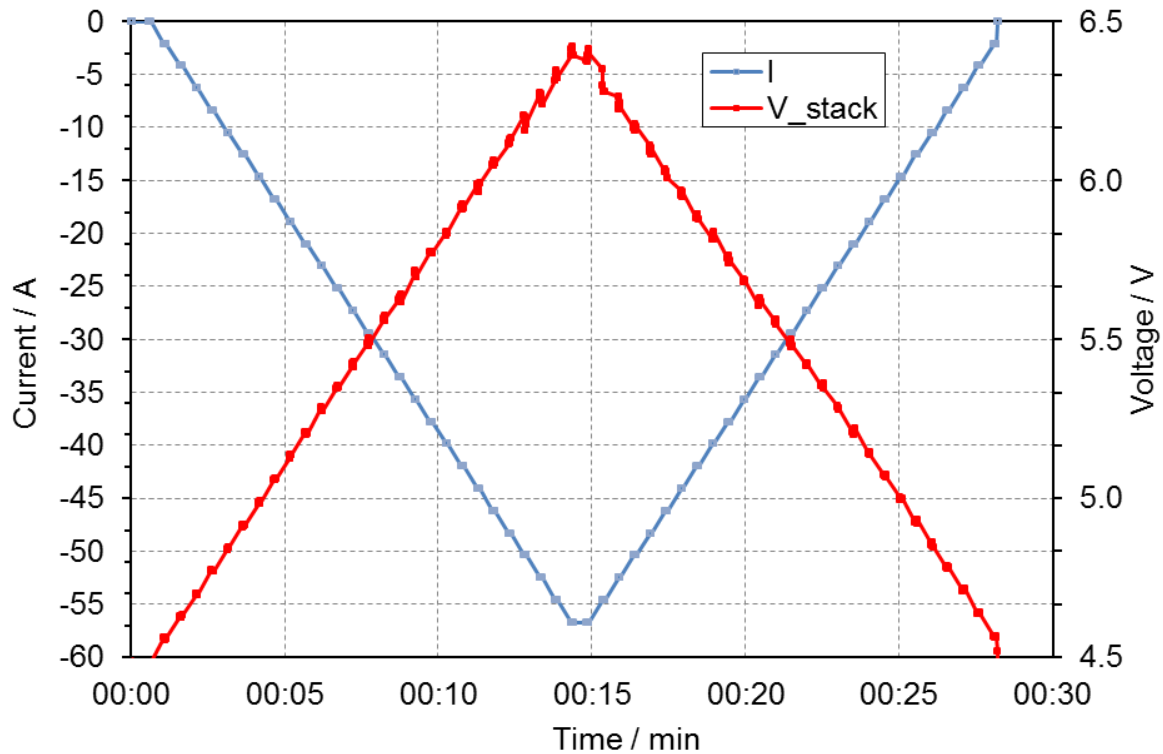


Figure 5: Example of TIP (current) and TOPs (SOEC stack voltage) recorded during j-V curve measurement (ascending and descending) showing their evolution.

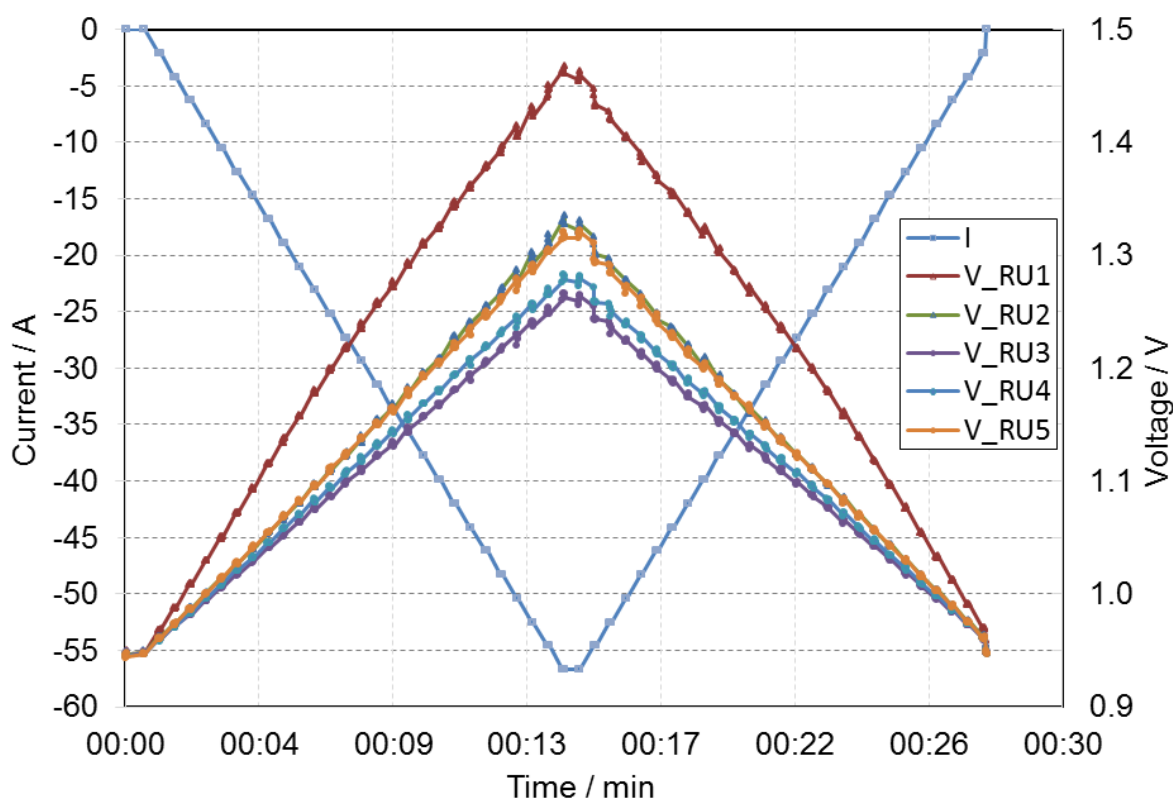


Figure 6: Example of TIP (current) and TOPs (SOEC RU voltages) recorded during j -V curve measurement (ascending and descending) showing their evolution.

7 Data Post Processing and Representation

Figures 7, 8, 9 and 10 below present examples of j -V curve measurements for SOFC (stack and RUs voltage) and SOEC (stack voltage). Figure 7 shows a j -V curve measurement (ascending and descending) for an SOFC stack with 5 repeating units (RUs) including its (top plate) temperature. This diagram displays the performance in terms of voltage at various current densities with hysteresis between the ascending and descending curves. The stack (top plate) temperature increases with increasing current density from 750 to 770°C due to the exothermal electrochemical reaction and the generated heat of the internal resistances of the repeat units (Joule heat). The reverse happens with subsequent decrease in current density.

Figure 8 shows the j -V curves (ascending part) of the 5 repeating units (RUs numbered RU1 to RU 5 from bottom to top) of an SOFC stack (see Figure 7) including its (top plate) temperature. This diagram gives information about the stack homogeneity. In this case the fifth RU (RU5) adjacent to the top plate of the stack shows the lowest performance at high current density. The stack temperature increases with increasing current density due to the exothermal electrochemical reaction and the generated heat of the internal resistances of the repeat units (Joule heat).

Figure 9 shows the descending parts of the j -V curves for the same RUs (see Figure 8) including stack (top plate) temperature. The different performances of the RUs in terms of voltage at various current densities can clearly be seen. Similar to the ascending part of the measurement, the fifth RU (RU5) shows the lowest performance at high current density. The stack (top plate) temperature decreases with decreasing current density due to the decreasing generated heat at lower current densities.

Figure 10 shows an example of a j - V curve (ascending and descending) of an SOEC stack including its (top plate) temperature. Due to the endothermic reaction the temperature remains nearly constant at 750°C. Rather minor deviations between the ascending and descending parts both in voltage and temperature are observed.

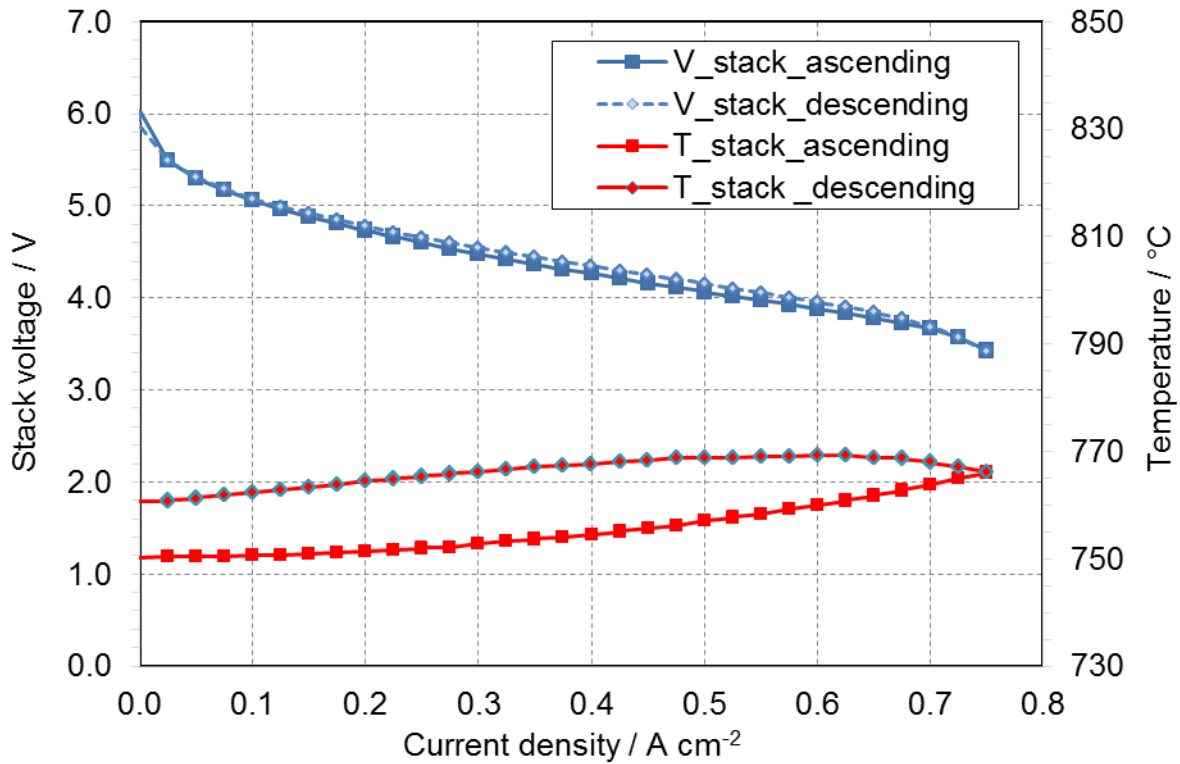


Figure 7: Example of a j - V curve measurement (ascending and descending) for an SOFC stack with 5 repeating units (RUs) including its (top plate) temperature

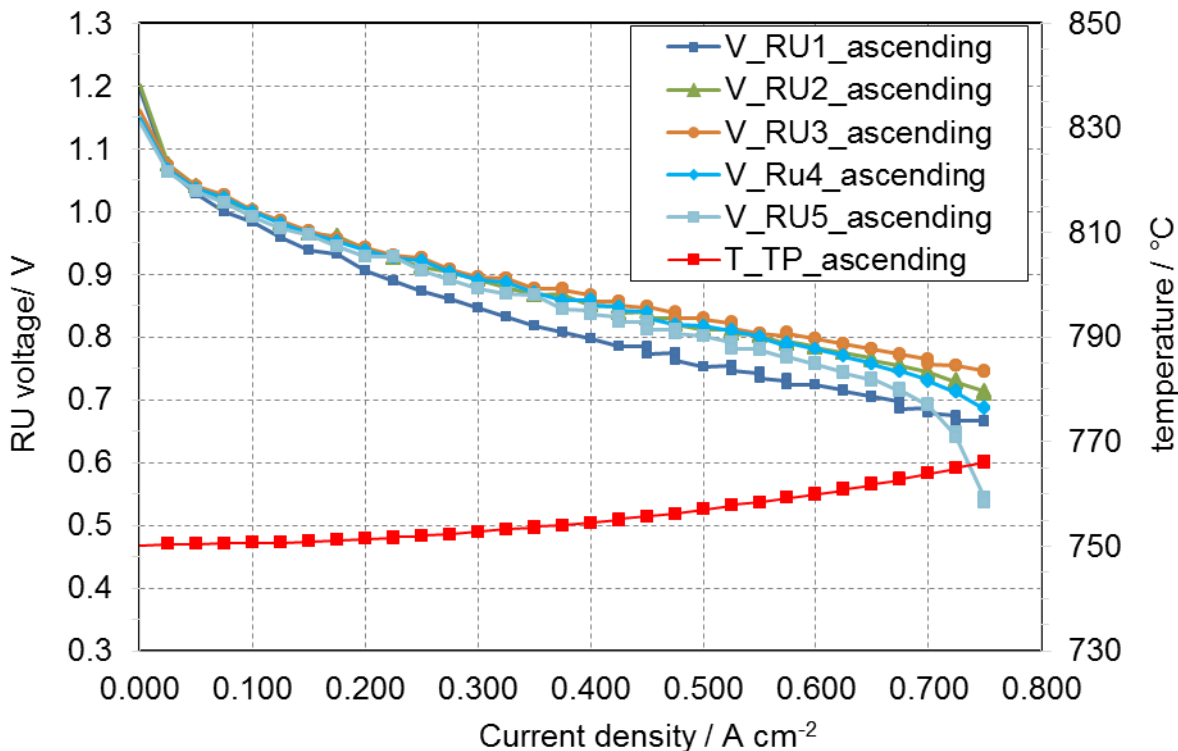


Figure 8: Example of the j-V curves (ascending parts) of the 5 repeating units (RUs numbered RU1 to RU 5 from bottom to top) of an SOFC stack (see Figure 7) including its (top plate) temperature

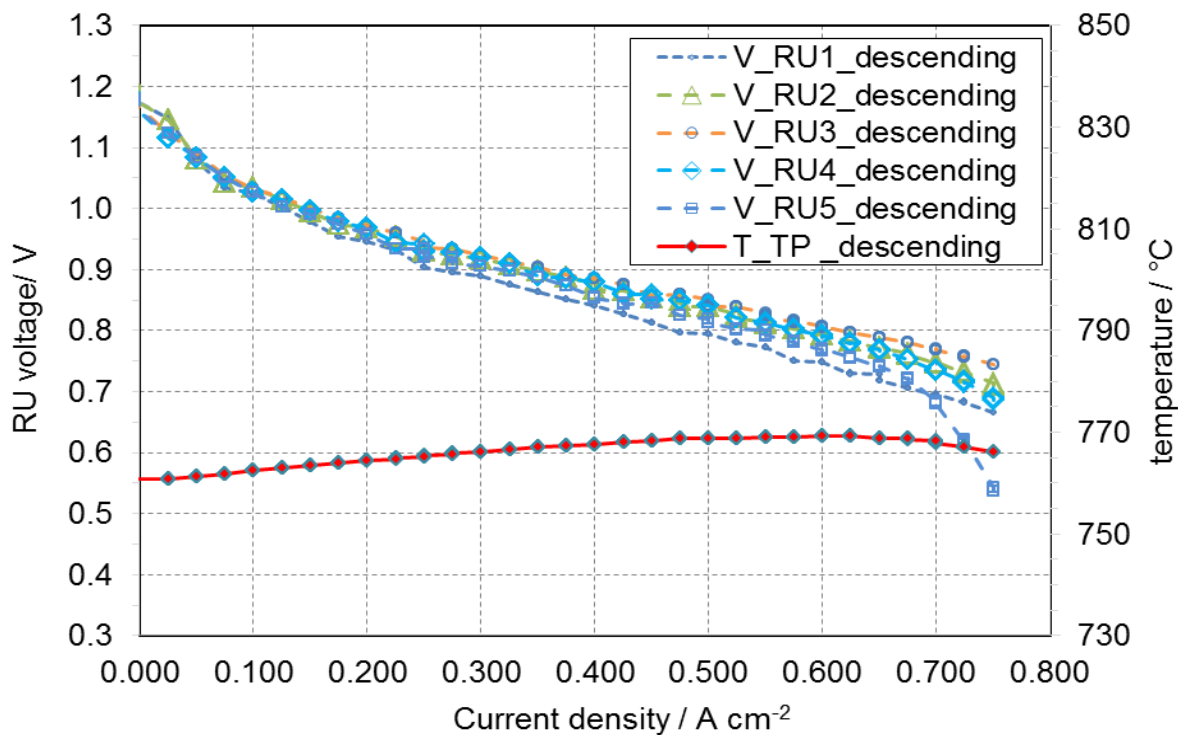


Figure 9: Example of the descending parts of the j-V curves for the 5 RUs (see Figure 8) including stack (top plate) temperature

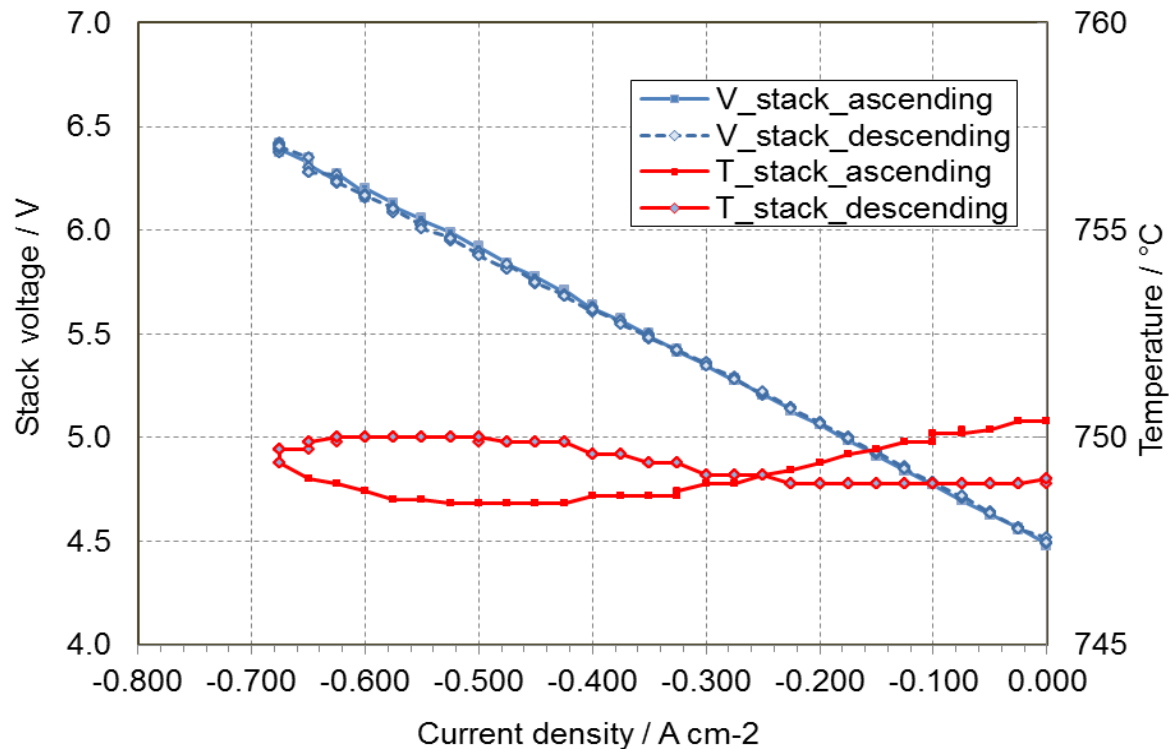


Figure 10: Example of a j - V curve measurement (ascending and descending) for an SOEC stack including its (top plate) temperature

8 Differences to Existing Procedures

This TM is nowadays rather common as shown by references found in literature [2-4]. Nevertheless, existing procedure as reference [2] does not specify a cut-off voltage to terminate test on current-voltage characteristic neither are all TIPs and TOPs mentioned. The FCTESQA procedures provide a detailed measurement method with a given set of current density steps. The present procedures also focuses on reproducibility of results (i.e. homogenous RU voltages) and explicitly define derived quantities, which is not the case for the existent procedures. Also, none of the existing procedures deals with SOEC whether at cell or stack level.

9 Bibliography

- [1] SOCTESQA-project document: "Test Module 00: General SOC Testing Guidelines", to be published on the homepage of the SOCTESQA project, <http://www.soctesqa.eu>
- [2] International Electrotechnical Commission (IEC) – Fuel Cell Technologies – Standard 62282 – Part 7-2: Single cell and stack test methods – Single cell and stack performance tests for solid oxide fuel cells (SOFC)
- [3] FCTESQA - Testing the voltage and power as function of current density (SOFC Stack), <https://ec.europa.eu/jrc/en/research-facility/fuel-cell-test-facility>.
- [4] FCTESQA - Testing the voltage and power as function of current density (Single Cells), <https://ec.europa.eu/jrc/en/research-facility/fuel-cell-test-facility>.