Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

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DELIVERABLE REPORT

D3.6 – FINAL DOCUMENT OF TEST PROTOCOLS	
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R	*Report*	*x*
P	*Prototype*	
D	*Demonstrator*	
O	*Other*	

SUMMARY

Keywords *Test protocol, generic test module, test program*

Abstract

The objective of the SOCTESQA project is to develop uniform and industry wide test procedures for high temperature solid oxide cells/stacks. These test procedures shall be applicable for different applications of energy systems integrating solid oxide cells/stacks, for instance SOFC-µCHP, SOFC-APU, SOEC for H_2 production (power-to-gas) and combined SOFC/SOEC for electricity storage (power-to-gas-to-power). The present document is the final compilation of 11 generic test modules developed in this project. These generic test modules can be combined to form application-specific test programs. All these test modules have been validated through extensive laboratory tests of SOC stacks by the project consortium. These individual test modules and test programs are public and can be downloaded from the project website (www.soctesqa.eu). Moreover, the guidelines and recommendations of these test modules are being transferred to standards developing organisations. In this context, based on the output of this project, an international standard (IEC 62282-8-101: Energy storage systems using fuel cell modules in reverse mode - Test procedures for solid oxide single cell and stack performance including operation in reverse mode) is currently under preparation.

Index

Abbreviations

APU	Auxiliary Power Unit
ASR	Area Specific Resistance
CHP	Combined Heat and Power
EIS	Electrochemical Impedance Spectroscopy
IEC	International Electrotechnical Commission
j-V	Current Density - Voltage
MCFC	Molten Carbonate Fuel Cell
PEMFC	Proton Exchange Membrane Fuel Cell
RU	Repeating Unit
SOC	Solid Oxide Cell
SOCTESQA	Solid Oxide Cell and Stack Testing, Safety and Quality Assurance
SOFC	Solid Oxide Fuel Cell
SOEC	Solid Oxide Electrolysis Cell

D3.6 – FINAL DOCUMENT OF TEST PROTOCOLS

1 Introduction

The objective of the SOCTESQA project is to develop uniform and industry wide test procedures for high temperature solid oxide cells/stacks. These test procedures shall be applicable for different applications of energy systems integrating solid oxide cells/stacks. While there are other applications of SOC (including SOFC and SOEC) technologies, the following four applications are considered in this project:

- Stationary SOFC application: µ-CHP and distributed power generation
- Mobile application of SOFC: SOFC-APU
- SOEC for H_2 production in a power-to-gas system
- Combined SOFC/SOEC for electricity storage in a power-to-gas-to-power system

For each application, a specific test program shall be defined to characterize the test object under typical operating conditions that are representative for the targeted application. A test program shall be composed of a series of sub-programs, which are called test modules, and each test module has its own test objective and contains specific procedures to be followed. In many cases, the same test module can be used in different test programs. Such a test module can then be called generic test module. Therefore, the main objective of the present project is to develop these generic test modules, which are basic elements of application-specific test programs. A combination of generic test modules in a meaningful series connection forms a test program.

At the beginning of the project, a test matrix, i.e. a list of generic test modules relevant for different applications, was defined based on a brief review of results from other relevant projects. For example, the project FCTESQA addressed cell/stack/system testing procedures for three types of fuel cells (PEMFC, SOFC and MCFC). Another project called STACKTEST dealt with testing procedures for PEMFC stacks. Moreover, industrial stake holders who develop SOFC/SOEC products were contacted to gather information regarding the required operation modes during the lifecycle of the product for each application.

During the running of the project, 11 generic test modules have been developed, including a master module (TM00) which deals with general issues regarding SOC testing. In order to check the applicability of the as-developed test modules, three rounds of test campaigns have been carried out by the project consortium (totally in 7 laboratories). All test laboratories have used short stacks with the same specifications and supplied by the same stack manufacturer, which enables results comparison with each other. Test modules have been reviewed and optimised after each round of test campaign.

The present document supplies the final compilation of 11 generic test modules. In addition, one test program related to the power-to-gas-to-power application is given as an example to illustrate how a test program can be built by combining different test modules. The guidelines and recommendations of these test modules are being transferred to standards developing organisations. Based on the output of this project, an international standard (IEC 62282-8-101: Energy storage systems using fuel cell modules in reverse mode - Test procedures for solid oxide single cell and stack performance including operation in reverse mode) is currently under preparation.

2 Test matrix

The following table gives a set of 18 possible test modules plus the master module TM00. The relevance of each test module for the four applications mentioned above is indicated. “x”/“-” means that the test module is relevant / irrelevant for the application. It should be noted that TM06 “Cyclic Voltammetry” is assigned as “irrelevant” for SOC characterization since this method is so far mainly used for the characterization of PEM fuel cell stacks. Nevertheless, it might become interesting for the SOC-characterization in the future. During the running of the SOCTESQA project, 11 modules (marked with bold texts) were able to be developed and validated through laboratory tests. Other modules were not developed mainly due to the foreseen difficulty in validating them using the existing testing capability of the project consortium.

Table 1: Test matrix for application-oriented testing

Generic Test Modules		Applications			
		Stationary SOFC-µCHP and distributed power generation	Mobile SOFC-APU	SOEC for H_2 production *power-to-gas*	Combined SOFC/SOEC for electricity storage *power-to-gas-to-power*
TM00	**General SOC Testing Guidelines**	x	x	x	x
TM01	Leakage Test	x	x	x	x
TM02	**Start-up**	x	x	x	x
TM03	**Current-voltage Characteristics**	x	x	x	x
TM04	**Electrochemical Impedance Spectroscopy**	x	x	x	x
TM05	Current Interruption	x	x	x	x
TM06	Cyclic Voltammetry	-	-	-	-
TM07	**Reactant Utilisation**	x	x	x	x
TM08	**Reactant Gas Composition**	x	x	x	x
TM09	**Temperature Sensitivity**	x	x	x	x
TM10	Pressure Sensitivity	-	-	x	x
TM11	Mechanical Load Sensitivity	x	x	x	x
TM12	**Operation under Constant Current**	x	x	x	x
TM13	**Operation under Varying Current**	x	x	x	x
TM14	**Thermal Cycling**	x	x	x	x
TM15	Redox Cycling	x	x	x	x
TM16	**Shut-down**	x	x	x	x
TM17	Vibration Test	-	x	-	-
TM18	Emergency Stop	x	x	x	x

3 Brief description of test modules

TM00: General SOC testing Guidelines

This master module gives general guidelines regarding the testing of solid oxide cells and stacks, covering terms and definitions, general safety aspects, test object and test system, list of quantities, useful equations etc. Unlike the other test modules (with numbers starting from 01), this test module will not be called by any application-specific test program. It addresses however common issues which are applicable for all other test modules.

TM01: Leakage Test

Gas-tightness is one of the most important requirements for an SOC stack. Insufficient gas-tightness leads to lower performance, lower efficiency and may create extra thermo-mechanical stress in the stack which shortens the stack lifetime. Leakage test can be performed in cold state (prior to the start-up and after the shut-down) or in hot state (during the stack operation). Both internal and external leakages are interesting to know. This test module defines the leakage test procedure.

TM02: Start-up

Start-up procedures are always needed for the test. First the cell/stack has to be heated up to the operation temperature. If the cell/stack is put into operation for the first time, reduction and conditioning will also be needed. In the case of APU-application, it might be necessary to draw current from the stack already during the heating-up phase in order to accelerate the temperature increase during the start-up process. Start-up parameters depend on the stack design and have to be given by stack manufacturers to avoid any damage.

TM03: Current-voltage Characteristics

Measuring the current-voltage curve is a basic method to characterize the performance of an SOC cell/stack. The main output will be the voltage of the stack as a function of current density. Current-voltage curves of all repeating units (RUs) in the stack can also be obtained if the voltages of RUs are measured separately. One test module from the previous project FCTESQA, Test Module TM SOFC ST M21 - “Polarisation curve for an SOFC Stack”, can be used as a reference for this test module. Key parameters for measuring a current-voltage curve such as the current variation rate, voltage limit as well as the way to present the data are defined. As the temperature of the stack usually changes while varying the current, it is recommended to plot also important temperature signals in the current-voltage curve plot.

TM04: Electrochemical Impedance Spectroscopy

While a current-voltage curve gives the performance and the overall internal resistance (often indicated by the so-called area specific resistance – *ASR*) of an RU or the whole stack, it does not supply detailed information on the different kinds of resistances of an RU. These resistances can be correlated e.g. to electron or ion transport, electrochemical reactions at the electrodes or gas transport processes within the cells. However, it is possible to use electrochemical impedance spectroscopy (EIS) to obtain this information. EIS is a very useful tool to identify the performance-limiting factor as well as the degradation mechanism. In this test module, the procedure to obtain the electrochemical impedance spectrum and the way to present the data are defined.

TM05: Current Interruption

The objective of the current interruption test is the determination of the ohmic resistance of an SOC stack or of an RU in the stack. The very fast change or interruption of the applied current induces a very fast or time independent variation in voltage due to the ohmic resistance and a slower or time dependent variation in voltage due to non-ohmic resistances. This module defines the procedure to perform current interruption test.

TM06: Cyclic Voltammetry (CV)

Although cyclic voltammetry has already been used in SOFC research for instance for the investigation of electrochemical reactions taking place at the electrode, it is not yet commonly used for testing SOC stacks. Instead, it is used for the characterization of PEM fuel cell stacks.

TM07: Reactant Utilization

The purpose of this test module is to measure the performance of an SOC stack as a function of reactant stoichiometry, which can be translated to fuel utilisation, air utilisation in fuel cell mode and steam/CO_2 conversion in electrolysis mode. This test will help to identify possible gas distribution or gas transport limitations in the stack.

TM08: Reactant Gas Composition

In this test module, the effect of the reactant gas composition on the performance of an SOC stack is determined. In SOFC mode, possible fuel-electrode gases can 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. Optionally reactant gas impurities, e.g. sulfur or poisoning hydrocarbons may be added. Depending on the application, different gas compositions may be relevant. On the oxidant side, the influence of O_2 concentration on the stack performance can be examined. In SOEC mode for the power-to-gas and the power-to-gas-to-power applications, the effect of the H_2 fraction (for instance in the range of 0.01-0.10) in the inlet gas can be determined.

TM09: Temperature Sensitivity

The purpose of this test module is to determine the temperature dependence of the stack performance. The stack operating temperature is often indicated by endplate temperatures or gas inlet/outlet temperatures and varied through the variation of the oven temperature.

TM10: Pressure Sensitivity

Operating the stack at elevated pressure can be beneficial for the SOEC operation since the generated H_2 may need to be stored in a compressed form. Energy consumption required for compression can be significantly reduced in this way. It is therefore necessary to determine the pressure dependence of the stack performance. Here the same pressure is meant for the fuel compartment, the oxidant compartment and the environment surrounding the stack. Some stacks may allow a big pressure difference between the fuel compartment and the environment and between the fuel compartment and the oxidant compartment. In this case, it may be possible to operate the stack with an elevated pressure only in the fuel compartment while the oxidant chamber and the environment are kept at atmospheric pressure.

TM11: Mechanical Load Sensitivity

Usually a certain level of compression force is needed in the stack to ensure good electrical contacts between cells and interconnects and/or sufficient gas-tightness. For stack testing, this compression force is often realized through applying a mechanical load onto the stack. The purpose of this test module is to determine the dependence of stack performance on the compression force.

TM12: Operation under Constant Current

In order to determine or predict the lifetime of an SOC stack, long-term endurance test under steady-state conditions is often conducted. The stack is usually operated in galvanostatic mode (constant current). The major output will be the degradation rate of the stack. In this test module, issues like the drift (?) of the stack temperature (at a fixed oven temperature) as a result of degradation need to be addressed. The way to extract and present the degradation rate is also defined. The operation conditions will have to be selected according to the application.

TM13: Operation under Varying Current

Many applications dictate a dynamic operation of the stack, varying load with time. In this test module, typical load profiles for each application are defined to simulate the real-life operation modes. As for the endurance test under constant load, the major output is the degradation rate.

TM14: Thermal Cycling

The capability to withstand a certain number of thermal cycles is an important requirement for an SOC-based system. In this module, conditions to perform thermal cycling of a stack are defined. Key parameters include temperature ramp rate, temperature range, atmosphere etc. The major output is degradation rate in terms of gas-tightness and performance upon thermal cycling.

TM15: Redox Cycling

In the case of fuel interruption, the fuel electrode will be subject to re-oxidation and subsequent reduction when fuel is restored. The capability of the stack to withstand redox cycling is another important requirement for an SOC-based system. This module defines procedures to perform redox cycling test.

TM16: Shut-down

Similar to the start-up module, general procedures of shut-down of the stack are described in this test module. Parameters like gas composition, cooling ramp rate should also be given by stack manufacturers to avoid any damage.

TM17: Vibration Test

Vibrational test can be important for the mobile application (SOFC-APU) to determine the tolerance of the stack to mechanical stresses induced by vibration of vehicles.

TM18: Emergency Stop

In certain circumstances, the system may be forced to stop urgently. The degradation of the stack after a certain number of “emergency stops” should therefore be evaluated. Here it is important to know what kinds of protection measures are implemented in the system to derive conditions of the stack.

4 Example of a test program

As mentioned above, a test program can be built through combining a number of generic test modules in a meaningful order. In this section, a test program related to the electricity storage application (power-to-gas-to-power) is given as an example.

For the electricity storage application, an SOC stack is operated in a combined SOFC/SOEC mode in order to convert steam to hydrogen with renewable power (SOEC steam electrolysis mode) when surplus electricity is available and produce electricity with hydrogen (SOFC mode) when electricity is again needed. In the test program described below, an application scenario of wind power storage was considered. A real wind power generation profile in relation to a real power demand profile was simplified and adapted to form an executable load profile of the SOC stack for the test. Here “executable” means the load profile can be easily realized by project partners (and later on by industrials) using their existing testing facilities. For this purpose, SOFC/SOEC operation cycles with a period of 24 h (20 h SOEC operation, 3 h SOFC operation and 1 h mode switching) were defined so that the mode switching could be done manually during the daytime.

Figure 1 shows the flow chart of the test program, in which 6 different test modules (TM02, TM03, TM04, TM12, TM13 and TM16) are integrated. After start-up (TM02), initial j-V (TM03) and EIS (TM04) characterizations in both SOFC and SOEC modes are performed, followed by pre-conditioning of the stack in SOFC mode for 120 hours through operation under constant current (TM12). Subsequently, the stack is operated under varying current (TM13) in combined SOFC/SOEC mode by following the simplified “wind profile” for around 500 hours. Prior to and after TM13, j-V (TM03) and EIS (TM04) characterizations are also performed to complete degradation analysis. The test program is finished with stack shut-down (TM16).

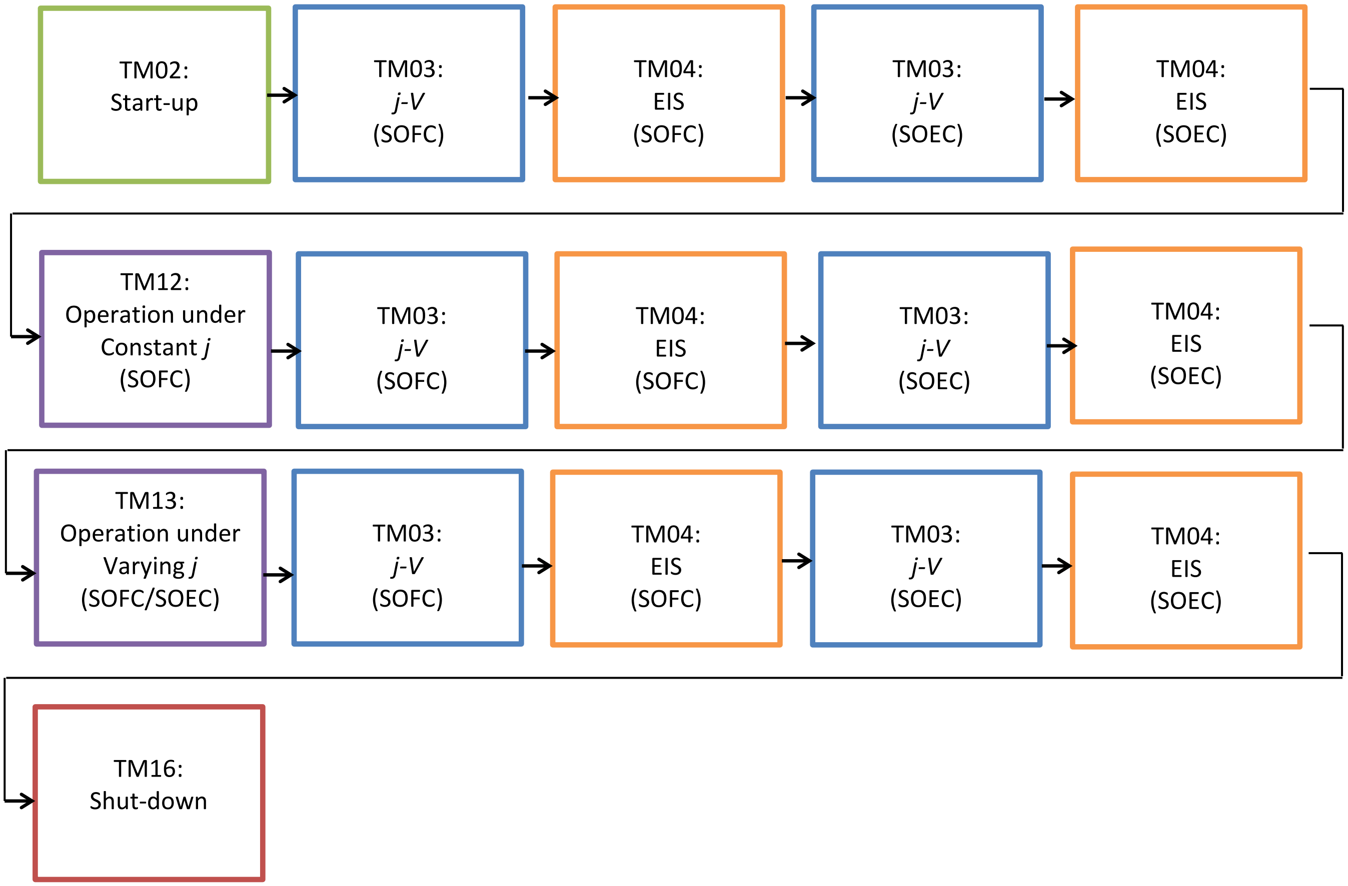

Figure 1: Flow chart of a test program for power-to-gas-to power application

Figure 2 and Figure 3 present schematic diagrams of current evolution as a function of time for test following wind profile and the overall test program.

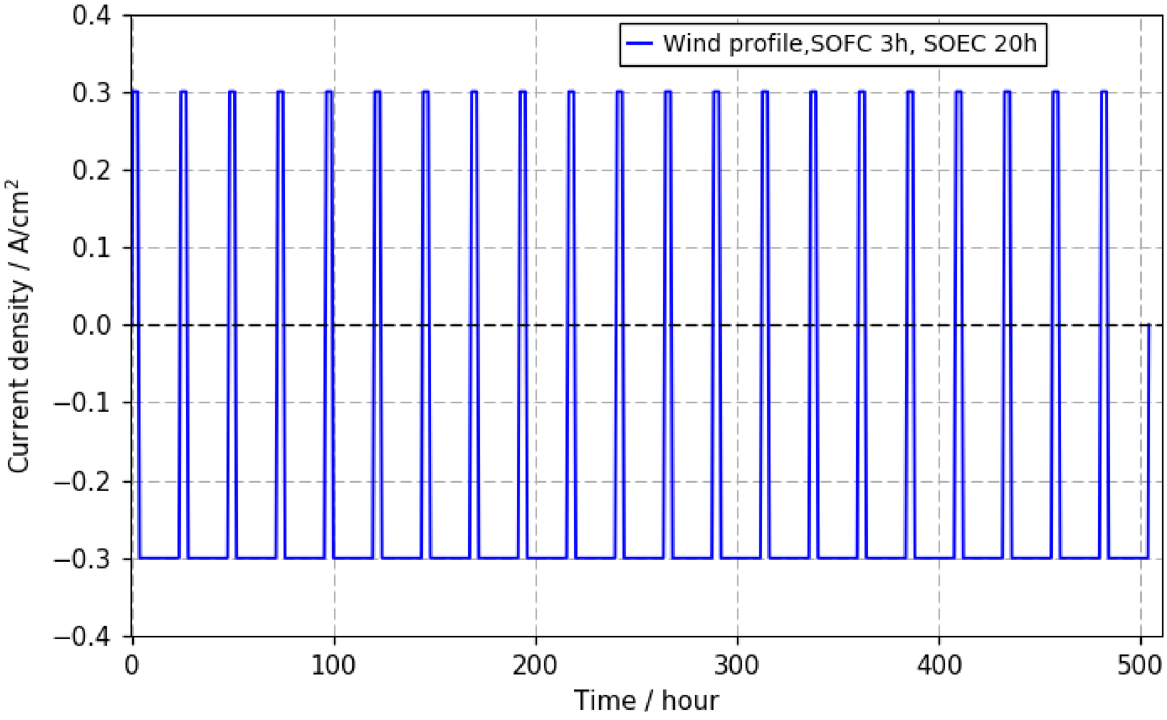

Figure 2: Schematic diagram of current evolution as a function of time for the test following wind profile

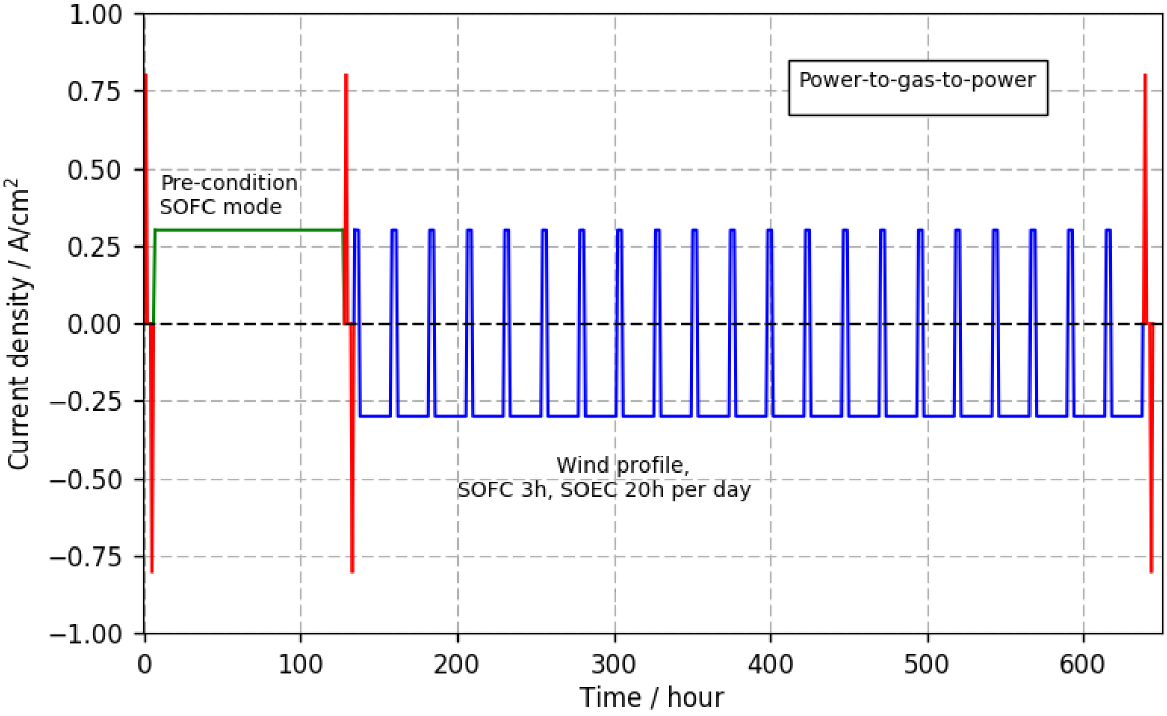

Figure 3: Schematic diagram of current evolution as a function of time for the overall test program

5 Appendices: Test modules

TM00: General SOC Testing Guidelines

TM02: Start-up

TM03: Current-voltage Characteristics

TM04: Electrochemical Impedance Spectroscopy

TM07: Reactant Utilisation

TM08: Reactant Gas Composition

TM09: Temperature Sensitivity

TM12: Operation under Constant Current

TM13: Operation under Varying Current

TM14: Thermal Cycling

TM16: Shut-down

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Test Module 00: General SOC Testing Guidelines

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Contents

Abbreviations

APU	Auxiliary power unit
ASR	Area specific resistance
BOP	Balance of plant
CHP	Combined heat and power
EIS	Electrochemical impedance spectroscopy
FFT	Fast Fourier Transform
HHV	Higher heating value
LHV	Lower heating value
nlpm	Normal litre per minute
OCV	Open circuit voltage
RMS	Root mean square
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
TOC	Test operating conditions
TOP	Test output parameter

TM00 – General SOC Testing Guidelines

1 Objective and scope

The present document shall give general guidelines regarding the testing of solid oxide cells and stacks. Unlike the other test modules of the SOCTESQA project (with numbers starting from 01), this test module (named TM00) will not be called by any test program which will be defined according to the targeted applications. It shall however address common issues which are applicable for all other test modules. It will therefore be referred to by the other test modules.

2 Normative references

The following documents have been considered for the present document:

IEC TS 62282-7-2:2014, Fuel cell technologies - Part 7-2: Single cell and stack test methods – Single cell and stack performance tests for solid oxide fuel cells (SOFC) [1]

IEC TS 62282-1:2013, Fuel cell technologies - Part 1: Terminology [2]

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

Table 1: Terms and Definitions

active electrode area (A)= effective electrode area	Geometric area of the electrode where the electrochemical reaction takes place. Area perpendicular to the direction of the current flow, usually expressed in m^2 or cm^2. NOTE: Usually this corresponds to the smaller area of the **negative electrode** and **positive electrode**.
area specific resistance (ASR)	Internal resistance of any component of a cell or a stack. It is normalized by the area and has a unit of Ω cm^2.
bipolar plate	Conductive plate separating individual cells in a stack, acting as current collector and gas distributor for the electrodes of the **SOC**. The **bipolar plate** usually incorporates flow field on both sides for the distribution of reactants and removal of products. The bipolar plate provides a physical barrier to avoid mixing of oxidant and fuel. The bipolar plate is also known as **interconnect**.
cell	A **cell** consists of two electrodes – a **positive electrode** and a

	negative electrode - with an electrolyte between them.
compression force (axial load)	Compressive load applied to the single **cell** or to the end plates of an **SOC** stack to assure electrical contact and/or gas tightness. NOTE: The compression force is in practice expressed in N.
conditioning	Related to the preliminary step of treatment that is required to properly operate an **SOC** and is usually realized by following a protocol specified by the manufacturer. NOTE: The conditioning may include reversible and/or irreversible processes depending on the cell technology.
contact layer	Layer applied between interconnect and the cell to minimize the contact resistance.
cross leakage	See **crossover**
crossover	Leakage between the positive electrode side and the negative electrode side of an **SOC**, in either direction, either through the electrolyte or through the sealant. NOTE: **crossover** is also called **cross leakage**.
current collector	Electronically conductive material in a cell that collects/conducts electrons from/to the electrodes.
current density	Current per unit **active electrode area** (usually expressed in A m^{-2} or A cm^{-2})
degradation rate	Rate at which a **cell**/**stack**'s performance deteriorates over time. Performance in this instance refers to the intended operation mode of the SOC (fuel production or electrical power generation). The degradation rate can be used to measure both recoverable and permanent losses in cell/stack performance. Depending on the operating mode, degradation rate can be expressed by the rate of evolution of any quantity of interest, which serves as an indicator of a **cell**/**stack**'s performance, for instance electrical current, fuel production rate, voltage, electrical power or area specific resistance. It is recommended to use always a sign before the number to indicate univocally the variation direction of the quantity. A positive sign means that the quantity is increasing with time and a negative sign means that the quantity is decreasing with time. Usually the degradation rate of a cell/stack is a function of time. Therefore the time frame under which the degradation rate is evaluated should always be indicated.
derived quantities	Values that can be derived or calculated from **test input parameters** and/or **test output parameters** (e.g. current density, reactant utilization, electrical efficiency). In comparison to **test output parameters**, **derived quantities** are not directly measurable.
external leakage	Leakage from gas compartments to the surrounding environment and

	vice versa.
efficiency	Efficiency is often defined as the ratio of useful output to total input.
electric efficiency	Ratio of net electric power produced by a fuel cell power system to the total enthalpy flow supplied to the fuel cell power system (based on LHV) [2].
heat recovery efficiency	Ratio of recovered heat flow of a fuel cell power system and the total enthalpy flow supplied to the fuel cell power system (based on LHV) [2].
heating value (HV)	The heating value of a fuel is the amount of heat released during the combustion of a specified amount of it. The heating value usually is the same as the thermodynamic heat of combustion $\Delta_f H$, or the thermodynamic heat of reaction $\Delta^r H$. The energy value is a characteristic for each substance. It is measured in unit of energy per unit of the substance, such as: kJ/kg or kJ/mol.
higher heating value (HHV)	The higher heating value (also known as gross calorific value or gross energy) of a fuel is defined as the amount of heat released by a specified quantity (initially at 25°C) once it is combusted and the products have returned to a temperature of 25°C, which takes into account the latent heat of vaporization of water in the combustion products. This is the same as the thermodynamic heat of combustion $\Delta_f H$ since the enthalpy change for the reaction assumes a common temperature of the compounds before and after combustion. Please note that HHV and LHV have positive signs whereas ΔH is negative.
interconnect	See **bipolar plate**
leakage current	Electric current in an unwanted conductive path other than a short-circuit
lower heating value (LHV)	The lower heating value of a fuel is defined as the amount of heat released by combusting a specified quantity (initially at 25°C) and returning the temperature of the combustion products to 150°C, which assumes the latent heat of vaporization of water in the reaction products is not recovered. The lower heating value is obtained by subtracting the latent heat of vaporization of the water vapour from the higher heating value.
negative electrode	Electrode at which fuel gas is consumed or produced. It may also be called fuel electrode. In the fuel cell mode, it is actually called anode where the fuel is oxidized. In the steam electrolysis mode, it is called cathode where steam is reduced producing H_2.
positive electrode	Electrode at which O_2 is consumed or produced. It may also be called oxidant electrode. In the fuel cell mode it is actually called cathode where O_2 is reduced producing oxide ions going through the

	electrolyte. In the steam electrolysis mode, it is called anode where two oxide ions are recombined to form one molecule of O_2.
protection gas (safety gas)	Usually refers to a mixture of hydrogen and inert gas (usually Ar or N_2). It is often used to protect the Ni-containing **negative electrode** of the **SOC** from being re-oxidized in case of abnormal operating conditions (e.g. fuel interruption, emergency stop of the test station). It can also be used during the heating-up and cooling down processes of the stack. The hydrogen fraction should be low enough (usually 0.03-0.05) to avoid the formation of explosive atmosphere. It should be high enough to keep a reducing atmosphere at the **negative electrode** of the **SOC** to avoid the re-oxidation of Ni.
rated current	Recommended continuous electric current as specified by the manufacturer, at which the **SOC** power system has been designed to operate
reactant utilization (U_{gas})	Gas utilization at the negative electrode (**fuel utilization in SOFC mode**, **steam conversion rate in SOEC mode**) or gas utilization at the positive electrode (air utilization or oxygen utilization in SOFC mode)
repeating unit (RU)	An elementary unit which periodically repeats itself to form a **stack**. It is composed of one single **cell** and two half-**interconnects** on both sides of the single **cell** and usually also sealant to assure gas-tightness and contact layers to minimize contact resistances between cells and **interconnects**.
reversible voltage /Nernst voltage(V_N)	Thermodynamic or reversible voltage of a redox system. This is a theoretical and not a measured value.
solid oxide cell (SOC)	**Cell** composed of three functional elements (negative electrode, electrolyte and positive electrode) based on ceramic oxide materials. Two electrodes made of ionic and/or electronic conducting ceramics are attached to one purely ionic conducting solid oxide ceramic electrolyte. Furthermore, the cell may contain a support layer which may be a ceramic layer or a non-ceramic layer as is the case for metal-supported cells. **SOC**s can be used as an **SOFC** or an **SOEC**.
solid oxide electrolysis cell (SOEC)	An **SOEC** is an **SOC** operated in the electrolysis mode, i.e., reversed fuel cell mode. It can be used to produce hydrogen from steam or to produce CO from CO_2. Electricity and also eventual heat are used as energy input.

solid oxide fuel cell (SOFC)	An **SOFC** is an **SOC** operated in the fuel cell mode. It is used for cogeneration of electricity and heat using fuels such as natural gas. In some applications only the electricity part is used and the co-generated heat is just dissipated.
stack	A fuel cell stack or an electrolyzer stack is composed of a number of **repeating units**. Normally two endplates (top plate and bottom plate) are used to facilitate applying a compression force to all **repeating units**.
test input parameter (TIP)	Parameters whose values can be set in order to define the test conditions of the test system including the operating conditions of the test object. TIPs have to be controllable and measurable. Values of TIPs are known before conducting the test. TIPs can be either static or variable. Static TIPs stay constant and variable TIPs are varied during the test duration of the selected test module.
test output parameter (TOP)	Parameters that indicate the response of the test system/test object as a result of variation of TIPs. Values of TOPs are unknown before conducting the test and will be measured during the test. TOPs must be measurable.
thermoneutral voltage (V_{tn})	Thermoneutral voltage relates to the enthalpy of a reaction. An electrolysis cell operates adiabatically at V_{tn}, i.e, no heat exchange is needed between the electrolyzer and its environment. In other words, heat produced in the cell due to the internal resistance (Joule heat) compensates exactly the heat needed by the endothermic electrolysis reaction. The electricity input to the electrolyzer matches exactly the enthalpy of the electrolysis reaction.
open circuit voltage (OCV)	The measured voltage of an electrochemical system without external electrical current.
voltage drop at the positive endplate ($V_{ep, pos}$)	Voltage drop due to the contact resistance between the positive endplate and its adjacent RU
voltage drop at the negative endplate ($V_{ep, neg}$)	Voltage drop due to the contact resistance between the negative endplate and its adjacent RU
voltage of a cell (V_{cell})	Voltage measured between the positive and negative electrode of a cell.
voltage of a RU (V_{RU})	Voltage measured between the two bipolar plates of a RU.
voltage of the stack (V_{stack})	The voltage measured between the two current connection terminals (for instance two endplates) of the stack. It should be noted that the stack voltage can be different from the sum of all RU voltages in the stack due to non-negligible voltage drops caused by contact resistances between endplates and their adjacent RUs.

4 General safety aspects

An operating SOC uses oxidizing and combustible gases. Typically, these gases are stored in high-pressure containers. The SOC itself may be operated at pressures greater than atmospheric pressure. The solid oxide cell/stack in particular operates at high temperatures, typically above 600°C, which are higher than the auto ignition temperatures of both hydrogen (565°C) and methane (537°C). Those who carry out solid oxide cell/stack assembly unit testing shall be trained and experienced in the operation of test systems and specifically in safety procedures involving explosive atmospheres, fire protection, electrical equipment and reactive, compressed gases.

The test personnel are responsible for obtaining and following all applicable safety regulations and generally accepted engineering practices related to their test system, facility, fuels (with particular attention to compressed gases), and exhaust products. Materials which are compatible with the use and storage of the reactant gases must be used during testing. Local safety codes and standards for working with hydrogen, hydrocarbons and carbon monoxide should be followed. In summary, safely operating a test station requires appropriate technical training and experience as well as safe facilities and equipment, all of which are outside the scope of this set of test procedures.

Nevertheless, a number of empirical guidelines can be given for good practice while carrying out the experimental tests, which can help identify potentially hazardous situations. Operating conditions of the SOC cell/stack assembly unit have to be carefully monitored as a means of preventing safety issues through an appropriate emergency shutdown system.

- Gas sensors: It is recommended to have gas sensors in close proximity to the test station (in particular for H_2, CO and CH_4), which are connected to an automated emergency shutdown system that cuts off fuel feed to the test station if hazardous concentrations of said gases are measured.

- Protection gas emergency supply: In the case of emergency shut-downs, it is recommended that a line containing an inert, reducing gas (“protection gas”, typically 3-5% hydrogen in a nitrogen matrix) be employed for the flushing of the negative electrode side of the solid oxide cell/stack assembly. This helps to expel flammable reactants from the system and at the same time protects the negative electrode (often composed of metallic nickel) from re-oxidizing, which may compromise the gas tightness of the SOC assembly.

- Protection gas for heating up and cooling down: Risk of explosion exists when the furnace temperature is below auto ignition temperatures of fuel gases in case of gas leakage. This risk can be avoided through the use of protection gas (typically 3-5% hydrogen in a nitrogen matrix) at the negative electrode side during phases of heating up and cooling down.The protection gas contains sufficiently low concentration of flammable gas to prevent explosion in case of gas leakage. It is yet still reducing to help to avoid re-oxidation of the negative electrode. It is recommended to use pre-mixed gases stored in bottles or big containers for this purpose. Caution should be taken when the protection gas is obtained through fresh mixing of two gas streams (H_2 and inert gas) in the test bench since mass flow controllers could fail, which might result in undesired concentration of H_2.

- Monitoring cell/stack voltage: A low open circuit voltage (OCV) may be an indication of gas leakage from the cell/stack assembly. During test operation (in closed circuit), it is advisable to monitor the cell/stack closed circuit voltage and implement an automated opening of the circuit in case the measured cell/stack voltage falls outside of the predefined operating window.

- Redundant Temperature sensors: It is recommended to place extra temperature sensors in the temperature-controlled furnace to ensure that igniting gas leaking from the cell/stack assembly may be detected from the measurement of high local temperatures.

- A humidity sensor could also be used at the oxidant outlet in order to detect cross leakage in the SOC cell/stack assembly unit.

- Pressure sensors could also help to control overpressures in the SOC cell/stack assembly unit and to prevent a break in tightness.

- When operating the SOC cell/stack in electrolysis mode, care must be taken that the produced hydrogen is adequately dealt with. For small production rates it will be sufficient to dilute the exhaust gas from the negative electrode with air or inert gas before venting. In case large stacks are used, it will be necessary to adopt a (catalytic) afterburner before venting.

This by no means claims to be an exhaustive or even sufficient list for safe operation during testing. It is therefore recommended to refer to applicable safety regulations and norms as well as consult experts in the field.

5 Test object

5.1 Cell

Figure 1 shows a single cell test object, as an assembly of two electrodes and the electrolyte which is integrated in a cell housing. For testing a cell ideally, the cell housing design and materials are optimized in that way of not having an influence on the results of the cell. Therefore, usually the electrical contact resistances of the electrodes in the cell housing are negligible and the supply of the electrodes with fuel and oxidant gases in the cell housing is ideal.

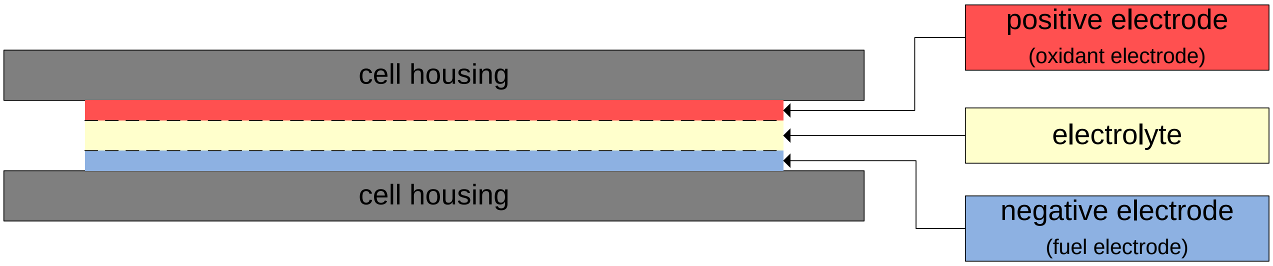

Figure 1: Scheme of a single cell test object consisting of an SOC in a cell housing

5.2 Stack

Figure 2 shows the scheme of an SOC-stack test object, consisting of multiple repeating units (RUs) which are assembled in series. The waved line in the middle shows that the number of repeating units is not specified. Electrical contacts and the gas distribution are provided by interconnects and the top/bottom plates. The stack results are generated by the repeating units, which might be influenced by electrical contact and gas flow conditions.

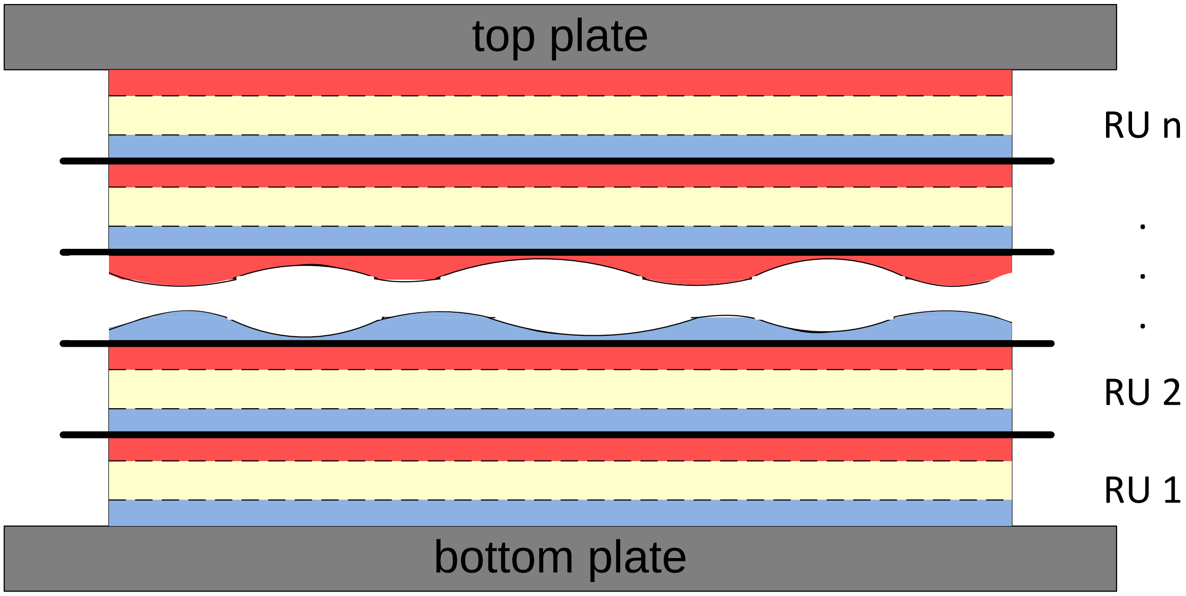

Figure 2: Scheme of an SOC stack test object

6 Test system

6.1 Subsystems in test system

Figure 3 shows a complete test system which consists of the solid oxide cell/stack assembly unit (test object) and the corresponding test and control subsystems. The test object is exposed to test inputs or operational conditions and delivers test outputs or object test results.

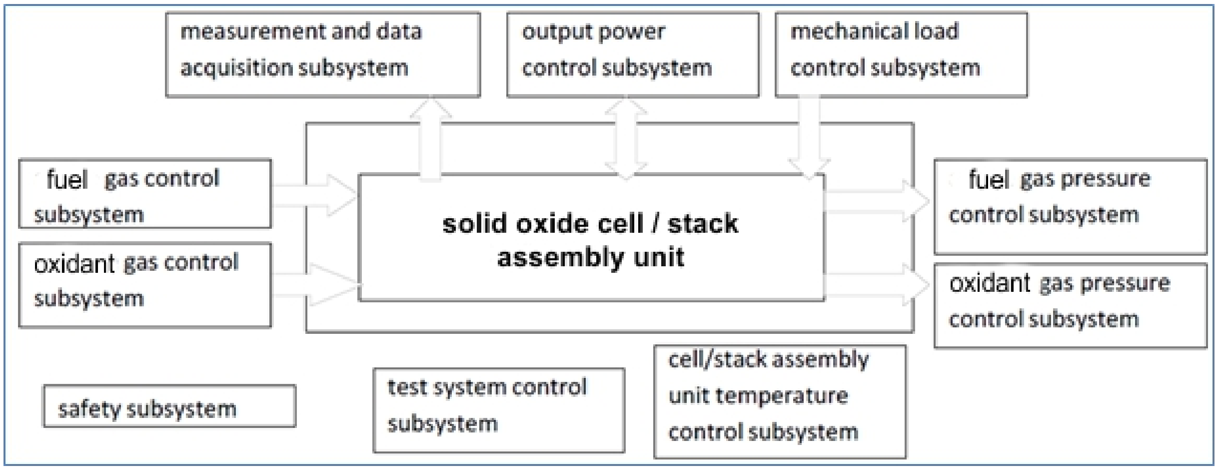

Figure 3: Schematic graph of a complete test system for high temperature solid oxide assembly unit

The subsystems can be classified according to their physical properties, which are of media, electrical, heat, mechanical and data type. Especially for high temperature solid oxide systems, the temperature control subsystem and the gas control subsystems (flow rate, pressure) play an important role for the test output results. Moreover, the complexity of the test system is significantly increased when the operation mode is changed from steady state operation to dynamic operating conditions.

6.2 Interface between test object and test environment

Figure 4 and Figure 5 show the physical interfaces between the SOC cell / SOC stack test object and the test environment. The physical interfaces between the test environment (test station) and the SOC cell/stack test object can be separated in media, electric, heat and mechanic interfaces. Media interfaces are the reactants inlet and outlet; electric interfaces are the load and power supply and voltage wiring; heat interface is the oven and the mechanic interface is to mount the cell/stack on the test station which may supply compression force to the cell/stack. These interfaces will be defined by different physical parameters. Depending on the test program these physical parameters can be either of input nature (TIP) or of output nature (TOP) (see Table 1).

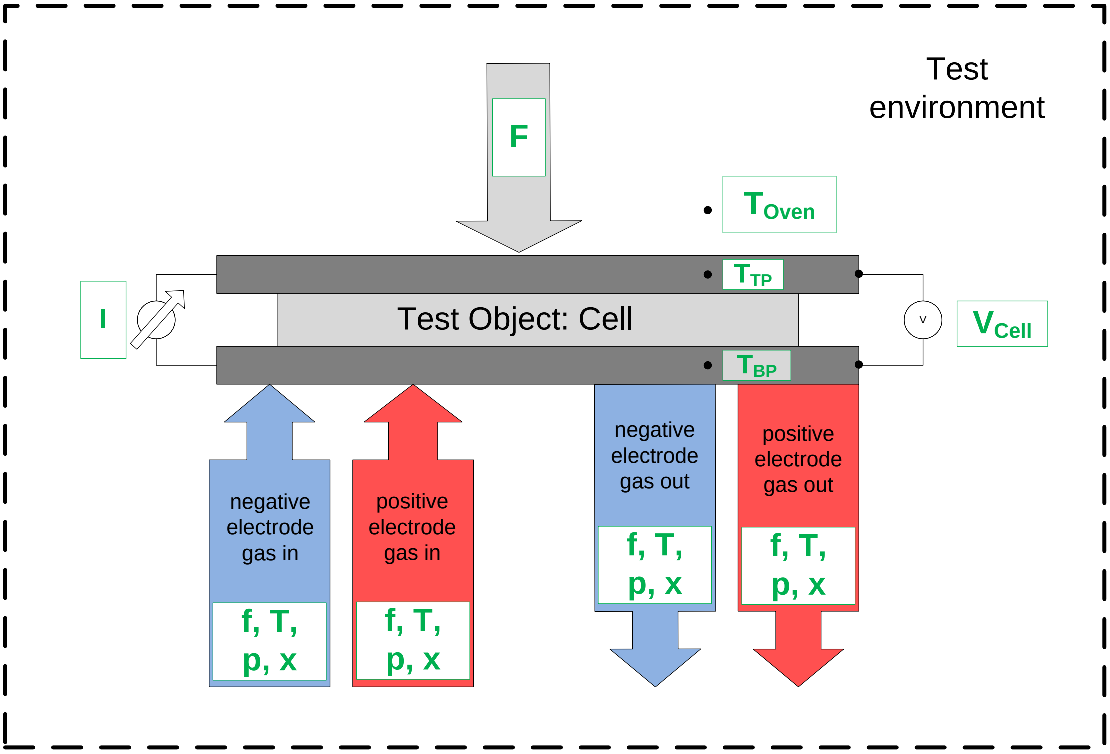

Figure 4: Interfaces between SOC cell and test environment

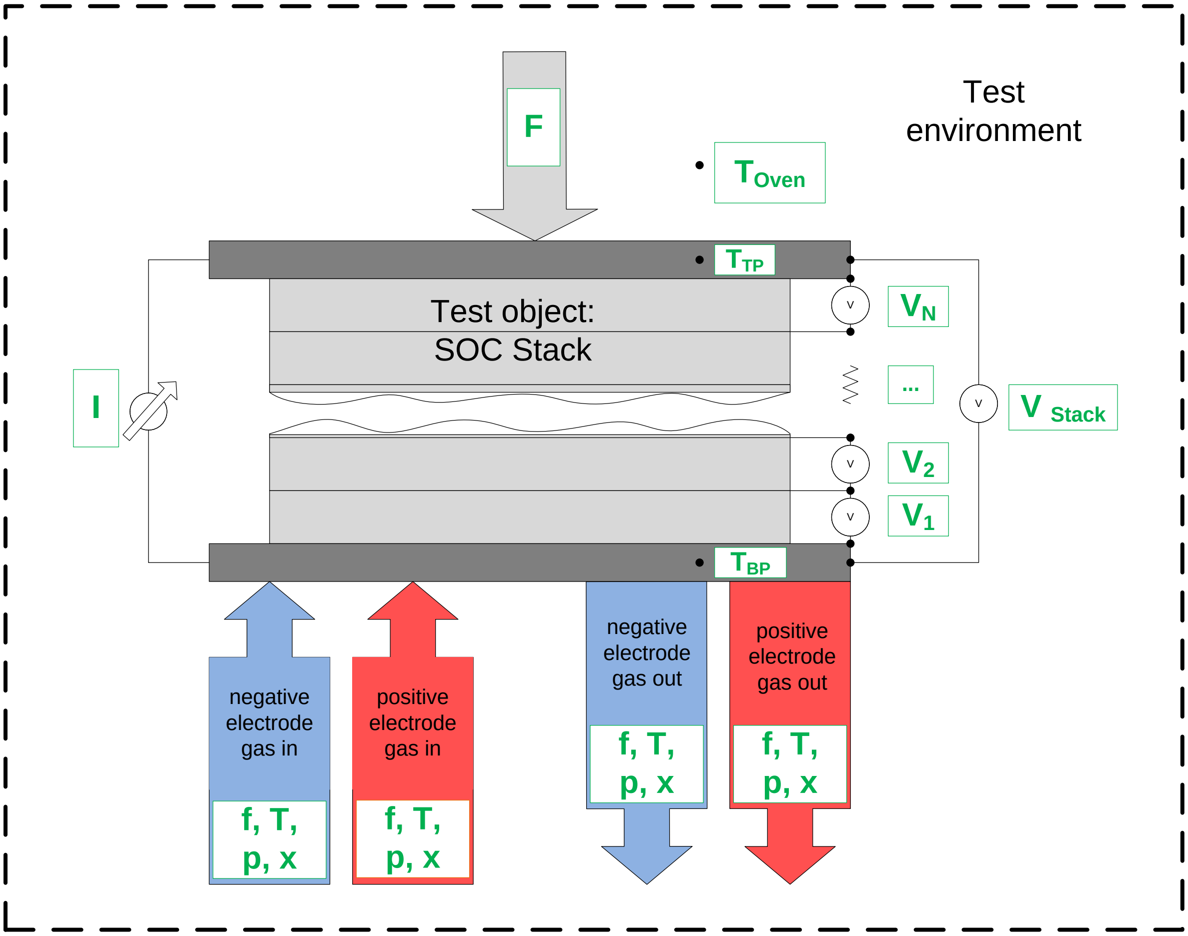

Figure 5: Interfaces between SOC stack and test environment

For the reactants at cell/stack inlet and/or outlet these parameters are:

- Temperature,*T*
- Flow (mass or volume),*f*
- Pressure,*p*
- Gas composition,*x*

The electric interface parameters are:

- Cell/stack current (common synonym is stack load), *I*
- Stack voltage, V_{stack}
- Voltages of cell or the individual repeating units, V_{cell} or $V_{RU,i}$

The heat interface parameters are:

- Oven temperature, T_{oven}
- Stack top plate temperature, T_{TP}
- Stack bottom plate temperature, T_{BP}
- Cell or stack internal temperature T_{cell} or $T_{stack,intern}$

Considering the fact that a temperature gradient in the stack is unavoidable and depends on various factors such as gas flow rates, current, thermal exchange rate with the furnace, it is difficult to determine the stack temperature. In practice, depending on the stack design, temperature sensors mounted at different locations in or around the stack (e.g., in endplates, in gas inlet/outlet channels, or just in close proximity to the stack) can be used to estimate the stack temperature. It is however important to identify the one that represents the best the real stack temperature. In order to make results comparable among different test facilities, it is necessary to agree prior to the test on which temperature is used to represent the stack temperature. For the results presentation, it is recommended to show also other temperatures measured in order to obtain information on the temperature distribution in the stack.

The mechanic interface parameter is:

- Cell or stack compression force, F_{compr}

Ideally, the positions for the setting/monitoring of the input/output parameters are as close as possible to the boundary of the test object and the test environment. Chapter 7 shows the recommended nomenclature to describe the parameters that are recommended to be measured and/or calculated.

6.3 Electrical output / input power control subsystem

The test objects 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. For *j-V* curves the electrical current normally is increased and decreased stepwise. This is usually done by connecting both current and voltage probes of the test object to an electronic load. Different setups are necessary for testing of either cells or stacks in fuel cell (SOFC) or electrolysis (SOEC) mode. 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 the repeat units of interest. The latter case has the advantage to examine the electrochemical behaviour of individual repeat units of the stack.

Setup for testing in SOFC mode:
In SOFC mode for testing of single cells or short stacks an additional voltage supply may be needed, e.g. to overcome voltage drops in the hot current wires (Fig. 6). The negative electrode in SOFC (anode) is connected with the negative pole of the electronic load. The power supply is connected with the electronic load in series in order to increase the voltage in the current circuit. For testing of SOC stacks, which have higher voltages than cells, usually the power supply is not needed (Fig. 7). In this case the stack is connected directly to the electronic load. If the voltage drop in the current circuit is too high, also an additional power supply is necessary for stack testing.

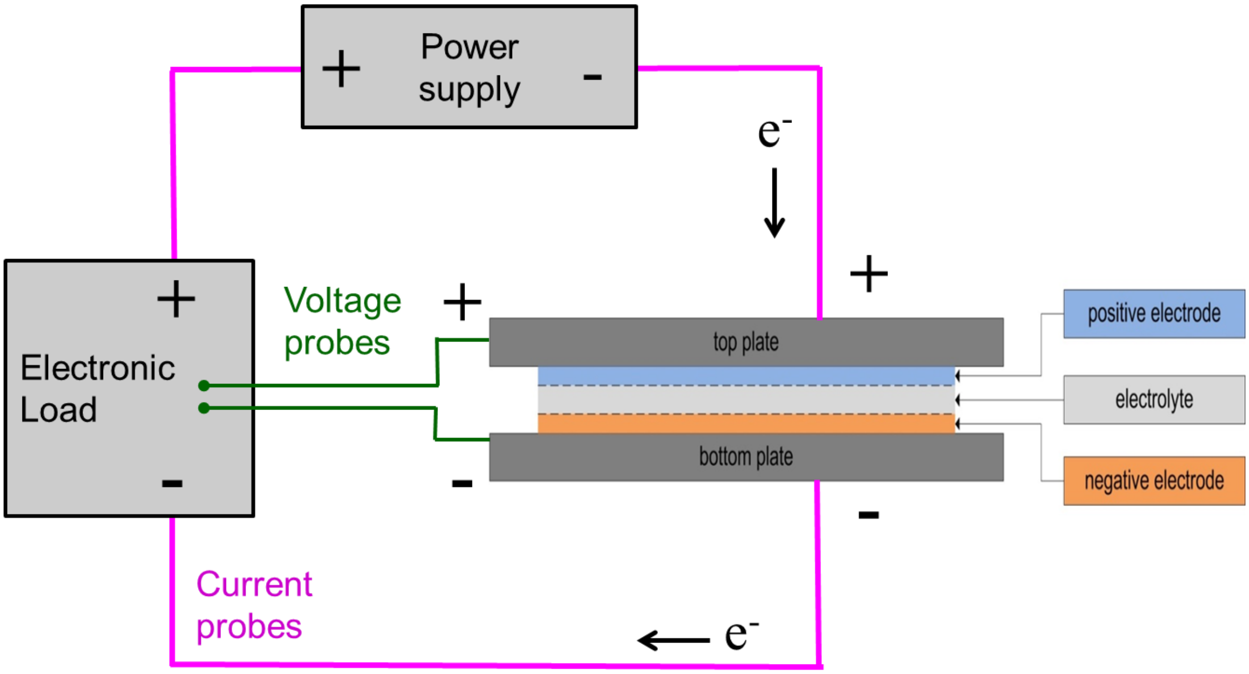

Figure 6: Setup of electrical power control system of SOC cells in SOFC mode

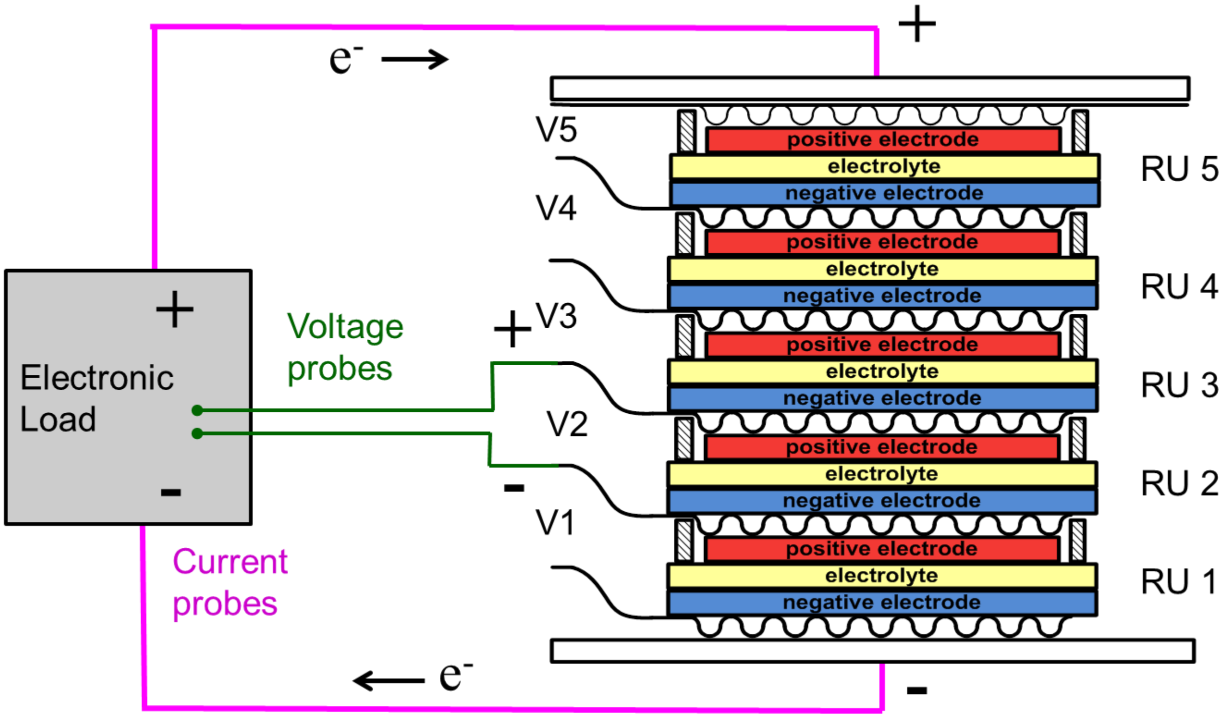

Figure 7: Setup of electrical power control system of SOC stacks in SOFC mode

Setup for testing in SOEC mode:

In SOEC mode a power supply in the current circuit is necessary in order to impose the electrolysis voltage on the SOEC cell/stack object (Fig. 8 and Fig. 9). 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. Therefore, the voltage of the power supply has to be higher than the voltage of the cell or stack.

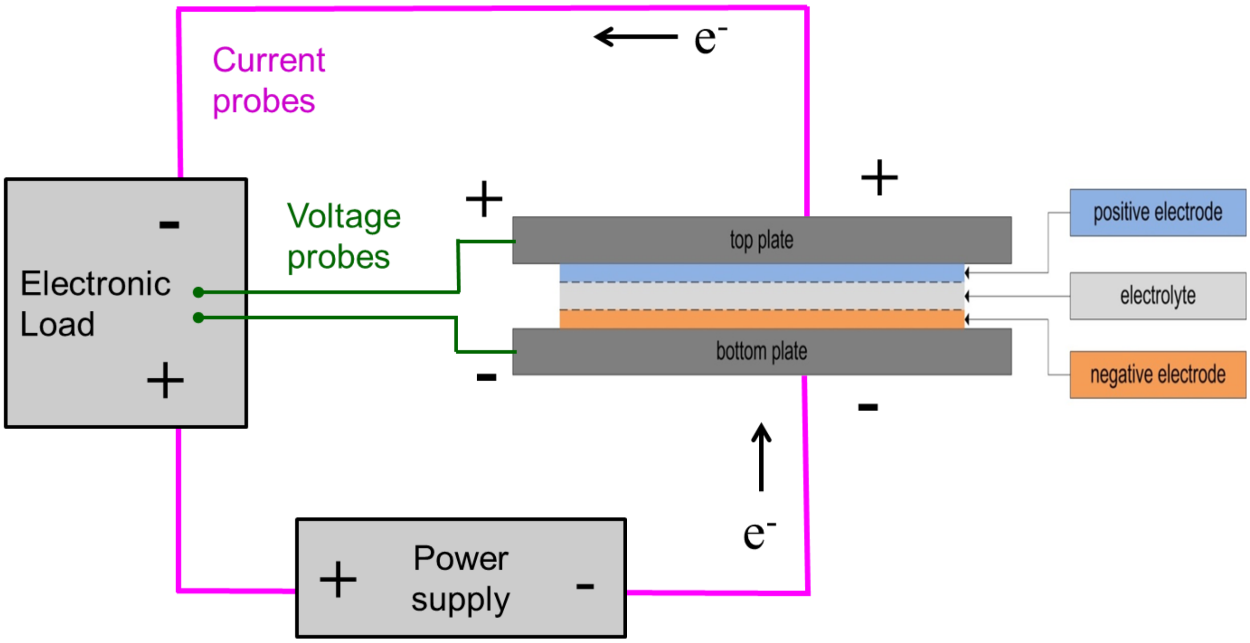

Figure 8: Setup of electrical power control system of SOC cells in SOEC mode

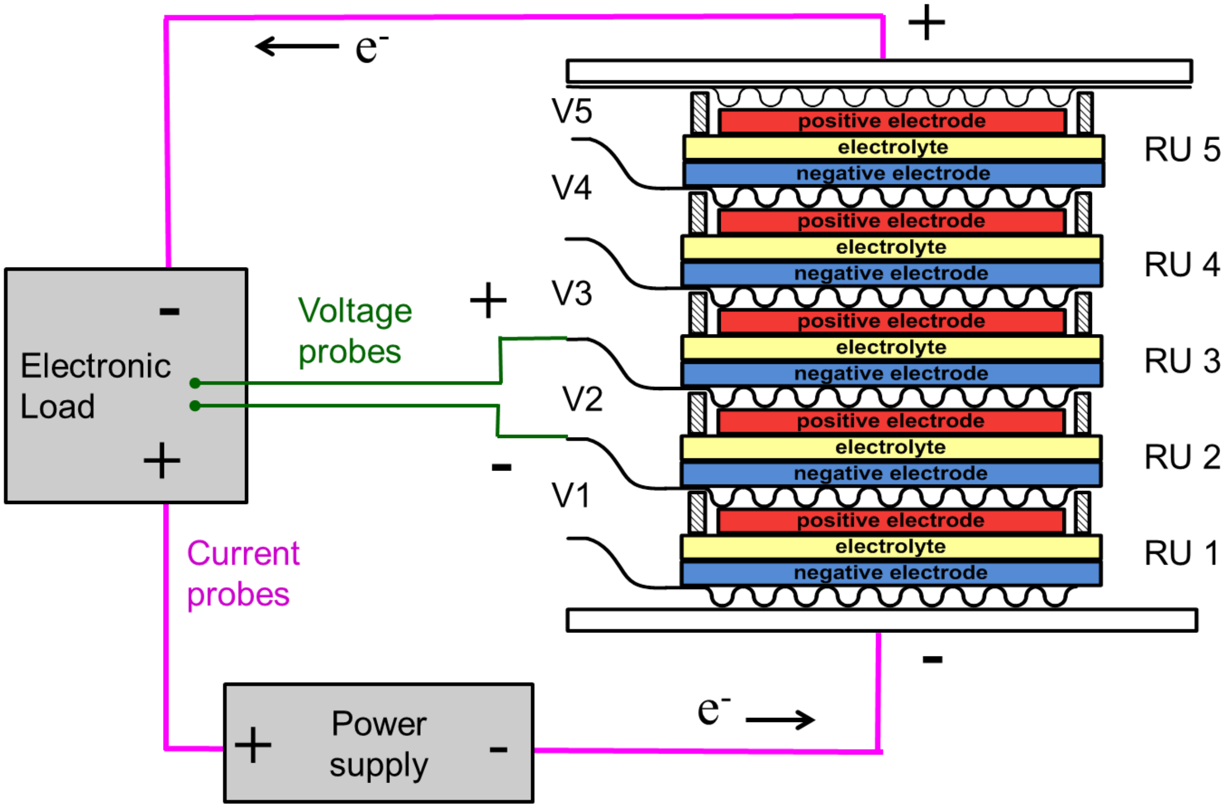

Figure 9: Setup of electrical power control system of SOC stacks in SOEC mode

6.4 Measurement methods of TIPs and TOPs and control accuracy

As mentioned in chapter 3, TIPs are controllable/regulated and measurable physical quantities whose values can be set in order to define the test conditions of the test system including the operating conditions of the test object. On the other side, TOPs are measurable physical quantities whose values are affected by the values of the TIPs. The process of determining the *precision*, *accuracy* and *uncertainty* of TIPs and TOPs measurement is an extensive process beyond the scope of this TM00.More details can be found in [3]: JCGM 100 (2008): Evaluation of measurement data - Guide to the expression of uncertainty in measurement (GUM).

Precision is the closeness of agreement between independent measurements of a quantity under the same conditions. It is a measure of how well a measurement can be made without reference to a theoretical or true value and depends only on the distribution of random errors.

Accuracy is the closeness of agreement between a measured value and the true value. Error is the difference between a measurement and the true value of the quantity being measured.

Uncertainty is the component of a reported value that characterizes the range of values within which the true value is asserted to lie. An uncertainty estimate should address error from all possible effects (both systematic and random) and, therefore, usually is the most appropriate means of expressing the accuracy of results.

Based on the aforementioned definitions it is clear that the most appropriate means of expressing the values of TOPs is the *Uncertainty* of the measured values, while for the TIPs, the *Control Accuracy* (which is usually instrument specific) should be reported.

Description of each testing parameter and measurement method follows:

Gas flow rate

Gas flow rates should be measured using mass flow meters. The mass flow meter size shall be selected taking into account the species of the measured gas and the flow range used during the test execution as well. Usually the digital mass flow meters used on the test benches are provided with an accuracy level of 1% of the Full Scale (maximum flow) and with a minimum measurable flow (generally 10% of the maximum flow). When using volumetric gas flow rate instead of mass flow rate, which is normally applied in the community of fuel cell testing, the conditions to which the gas flow rate refers should be carefully checked (see the note in chapter 7).

Reactant gas temperature

Reactant gas temperature shall be measured at inlet and outlet of the cell/stack. The measurement shall be done by using thermocouples placed in the gas stream line and as near as possible to the object under test.

Reactant gas pressure

Pressures of reactant gases shall be measured upstream (at the cell/stack gas inlet lines) at both positive and negative electrodes gas lines. The pressure shall be measured by using calibrated pressure sensors. Caution shall be taken to minimize pressure losses in the piping from the cell/stack and the pressure sensors. In the test report the selected option shall be stated, whether the cell/stack inlet pressure or the cell/stack outlet pressure is controlled to be kept constant. However, cell/stack inlet pressure control is strongly recommended.

Voltage

Voltage probes shall be placed as close as possible to the cell/stack terminals to prevent that the measure is affected by losses on the cables. Voltage losses might be relevant at high temperature.

Current

Electrical current shall be measured directly by the electrical load in SOFC mode or power supply in SOEC mode. Current can also be measured by using a shunt on the power cables.

The following table contains all control accuracies for each TIP while for each TOP the instrument uncertainty is stated.

The measuring parameters listed in Table 2 should be recorded and stored with a sampling rate fast enough to ensure that all relevant changes in the parameters are recorded for later data analyses. The required sampling rate depends on the test objective. In most cases a sampling rate of 1 Hz is considered sufficient.

Table 2: Control accuracies for each TIP and instrument uncertainty of TOPs

Symbol	Instrument Uncertainty	Control Accuracy
I	± 1% *	± 0.001A
V	± 0.5%*	± 0.001V**
T	± 1 ºC	± 2 ºC
$f_{neg/pos,\ in/out}$	± 2% ***	± 2% FS (Full scale)
x_i	± 2.0 mol % for H_2, H_2O,N_2; ± 1.0 mol % for CO, CO_2, CH_4	± 2.0 mol % for H_2, H_2O, N_2; ± 2.0 mol % for CO, CO_2, CH_4
x_{O2}	± 0.3 mol % O_2	± 1.0 mol % O_2
$p_{neg/pos,\ in/out}$	± 3%***	± 2%

** Note 1: Percentages relative to setpoint.*
*** Note 2: Potentiostatic testing*
**** Note 3: Percentage of maximum expected value.*

6.5 Stability criteria

To evaluate the influence of operating conditions on the cell/stack performance, a certain degree of stability of both TIPs and TOPs is required. One simple option to evaluate stability is to record a parameter with a desired sampling rate for a desired sampling duration. The parameter is considered stable if the maximum variation (difference between the maximum value and the minimum value recorded) does not exceed a pre-defined threshold value. However, it is difficult to give a generic definition of the sampling rate, sampling duration and variation threshold. This is due to the fact that each individual TIP may have a different impact on the cell/stack performance. If the cell/stack performance is not so sensitive to a TIP, it is then acceptable to set a larger variation threshold for that TIP. A parameter sensitivity investigation needs thus to be performed to define precisely the stability criteria. In addition, the measurement uncertainty of TOPs should also be taken into account for the definition of stability criteria. Furthermore, in many cases a compromise needs to be found between good stability and reasonable test duration. A very strict stability criterion may not be fulfilled due to the time constraint.

Nevertheless, the following stability criteria in Table 3 can be taken as a reference and can be adapted according to the test object, test system and test objective.

Table 3: Stability criteria for TIPs and TOPs as a reference.

Parameter	Sampling rate	Sampling duration	Variation threshold
V_{cell}, $V_{RU,i}$	1 Hz	3 min	5 mV (SOFC mode) 10 mV (SOEC mode)
I	1 Hz	3 min	0.5 A
T	1 Hz	3 min	1 K
$f_{neg/pos,\ in/out}$	1 Hz	3 min	3% FS
$p_{neg/pos,\ in/out}$	1 Hz	3 min	3% FS

6.6 Mounting of the test object into the test system

When mounting the test object into the test system some test-related factors should be considered in addition to the general safety precautions of the test system.

When testing a **single ceramic solid oxide cell**, the cell is mounted within a cell test fixture, which provides gas connections and possible sensory connections similar to a stack assembly. When mounting a cell within a cell test fixture, some general considerations have to be made to assure that the influence of the test setup on the results is minimized:

- Electrical contact to both the negative and positive electrodes has to be assured; this is done by ensuring actual mechanical contact of the cell to the contact components, and by having sufficient contact points to avoid current restrictions.
- A well-defined or masked geometric area of an electrode has to be defined as the geometric cell area.
- The cell has to be sufficiently leak-tight to avoid significant mixing of the test gases with other gases, before passing the negative or positive electrodes.
- Any potential pollutants (sulphur, phosphorous, etc.) from components used in the cell test fixture have to be avoided.

- Mechanical stresses from the cell test fixture components have to be avoided, to prevent any potential cracking of the cell during testing.
- Furthermore, flow fields at the electrodes have to distribute the gas evenly, and without a potential turbulent flow, to avoid gas starvation and mass transport limitations at the electrodes.

When the above mentioned considerations are adequately taken care of, the cell test fixture can be handled similar to a stack assembly.

When the **cell/stack assembly** has to be mounted in the test setup, it has to be securely positioned, to avoid accidental movement during testing. Electrical connections have to be properly done and gas connections securely tightened, while sharp bends of the gas tubes should be avoided. Isolation resistances between the repeating units in the stack should be checked to avoid a short circuit. Gas tightness of the gas supply subsystem of the test station, of the stack itself and of the interface between stack and test station should also be checked. Furthermore, the gases fed into the cell test fixture/stack assembly have to be preheated to the ambient oven temperature, to avoid a significant temperature difference at the gas inlet. The mechanical load applied on the cell/stack should be uniform. In some cases, the compression mechanism required for stack transport may need to be removed after mounting the stack into the test station.

6.7 Precaution regarding the quality of the testing environment

Following the recommended practices described in this test module on mounting of the stack in the test bench/setup is advised, and any deviations from described practices should be noted in the test report. Further, the responsible organization shall make sure the quality of the testing environment is as good as possible, with emphasis on accuracy of measurements and controls, e.g. that mass flow controllers are correctly controlled and that the testing environment elsewise is functioning as intended.

7 List of quantities

Table 4: List of quantities

Description of quantity	Symbol	Unit often used	SI Unit	Unit conversion
Active electrode area	A	cm²	m²	1 m² = 10^4 cm²
Area specific resistance	ASR	Ω cm²	Ω m²	1 Ω m² = 10^4 Ω cm²
Phase angle	φ	°	rad	$1° = \frac{\pi}{180} rad$
Compression force applied onto the cell/stack	F_{compr}	N	N	-
Capacitance of positive electrode	C_{pos}	F	F	-
Capacitance of negative electrode	C_{neg}	F	F	-
Flow rate of component *i* in the negative	$f_{i,neg,in}$	nlpm, slpm	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$

electrode gas stream at cell/stack inlet		l_n min^{-1}, l_s min^{-1}		
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}	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$
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}	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$
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}	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$
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}	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$
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}	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$
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}	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$
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}	$m^3 s^{-1}$	1 lpm = $10^{-4}/6$ $m^3 s^{-1}$
Gibbs free enthalpy of reaction	$\Delta^r G$	kJ/mol	J/mol	1 kJ/mol = 10^3 J/mol
Enthalpy of reaction	$\Delta^r H$	kJ/mol	J/mol	1 kJ/mol = 10^3 J/mol
Higher heating value of fuel component *i*	HHV_i	J mol^{-1}	J mol^{-1}	-
Electrical current through the cell/stack	I	A	A	-
Inductance	L	H	H	-
Amplitude of alternating current	$\bar{I}$	A	A	-
Imaginary unit	i	-	-	-
Electrical current density through the cell/stack	j	A cm^{-2}	A m^{-2}	1 A cm^{-2} = 10^4 A m^{-2}
Lower heating value of fuel component *i*	LHV_i	J mol^{-1}	J mol^{-1}	-

Number of measuring periods for each measuring point for EIS spectra	k	-	-	-
Number of cycles	m	-	-	-
Number of repeating units in the stack	N	-	-	-
Number of measuring points per decade for EIS spectra	n	-	-	-
Electrical efficiency based on LHV of fuels	$\eta_{el,LHV}$	-	-	-
Electrical efficiency based on HHV of fuels	$\eta_{el,HHV}$	-	-	-
Electrical power density (area specific)	$P_{d,el}$	W cm^{-2}	J s^{-1} m^{-2}	1 W cm^{-2} = 10^{4} W m^{-2}
Electrical power of the cell/stack	P_{el}	W	J s^{-1} (W)	-
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)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
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)	1 kPa = 10^{3} Pa 1 mbar = 100 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)	1 kPa = 10^{3} Pa 1 mbar = 100 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)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
Pressure of the negative electrode gas stream at cell/stack inlet	$p_{neg,in}$	mbar, kPa	N m^{-2} (Pa)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
Pressure of the negative electrode gas stream at cell/stack outlet	$p_{neg,out}$	mbar, kPa	N m^{-2} (Pa)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
Pressure of the positive electrode gas stream at cell/stack inlet	$p_{pos,in}$	mbar, kPa	N m^{-2} (Pa)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
Pressure of the positive electrode gas stream at cell/stack outlet	$p_{pos,out}$	mbar, kPa	N m^{-2} (Pa)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
Pressure drop of the negative electrode gas stream over the cell/stack	Δp_{neg}	mbar, kPa	N m^{-2} (Pa)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
Pressure drop of the positive electrode gas stream over the cell/stack	Δp_{pos}	mbar, kPa	N m^{-2} (Pa)	1 kPa = 10^{3} Pa 1 mbar = 100 Pa
Electrical charge	Q	C	C	-

Resistance	R	Ω	Ω	-
Low frequency resistance	R_{LF}	Ω	Ω	-
Total (or overall) resistance	R_{tot}	Ω	Ω	-
High frequency resistance	R_{HF}	Ω	Ω	-
Ohmic resistance	R_{ohm}	Ω	Ω	-
Polarisation resistance of positive electrode	$R_{pol,pos}$	Ω	Ω	-
Polarisation resistance of negative electrode	$R_{pol,neg}$	Ω	Ω	-
Gas concentration resistance of positive electrode	$R_{GC,pos}$	Ω	Ω	-
Gas concentration resistance of negative electrode	$R_{GC,neg}$	Ω	Ω	-
Time	t	s, min, h	s	1 min = 60 s 1 h = 3600 s
Average temperature of the stack	T_{av}	°C	K	x °C = (x + 273.15) K
Temperature of the bottom plate of the stack	T_{BP}	°C	K	x °C = (x + 273.15) K
Temperature of the cell	T_{cell}	°C	K	x °C = (x + 273.15) K
Maximum temperature difference during start-up	ΔT_{max}	°C	K	x °C = (x + 273.15) K
Temperature of the negative electrode gas stream at cell/stack inlet	$T_{neg,in}$	°C	K	x °C = (x + 273.15) K
Temperature of the negative electrode gas stream at cell/stack outlet	$T_{neg,out}$	°C	K	x °C = (x + 273.15) K
Temperature of the pre-heater for preheating the negative electrode gas stream	$T_{PH,neg}$	°C	K	x °C = (x + 273.15) K
Temperature of the pre-heater for preheating the positive electrode gas stream	$T_{PH,pos}$	°C	K	x °C = (x + 273.15) K
Temperature of the positive electrode gas stream at cell/stack inlet	$T_{pos,in}$	°C	K	x °C = (x + 273.15) K
Temperature of the positive electrode gas stream at cell/stack outlet	$T_{pos,out}$	°C	K	x °C = (x + 273.15) K
Stack temperature	T_{stack}	°C	K	x °C = (x + 273.15) K

Temperature of the top plate of the stack	T_{TP}	°C	K	x °C = (x + 273.15) K
Temperature of the oven	T_{oven}	°C	K	x °C = (x + 273.15) K
Frequency	υ	Hz	Hz	-
Minimum frequency for EIS spectra	υ_{min}	Hz	Hz	-
Maximum frequency for EIS spectra	υ_{max}	Hz	Hz	-
Gas utilization at the negative electrode (fuel utilization in SOFC mode, steam conversion in SOEC mode)	$U_{gas,neg}$	%	-	-
Gas utilization at the positive electrode (air utilization or oxygen utilization)	$U_{gas,pos}$	%	-	-
Volume	v	cm^3	m^3	1 $cm^3 = 10^{-6}$ m^3
Amplitude of alternating voltage	$\bar{V}$	V	V	-
Voltage of the cell	V_{cell}	V	V	-
Voltage drop at the positive endplate	$V_{ep,pos}$	V	V	
Voltage drop at the negative endplate	$V_{ep,neg}$	V	V	
Reversible (thermodynamic) voltage / Nernst voltage	V_N	V	V	-
Average RU voltage of all RUs in the stack	$V_{RU,av}$	V	V	-
Voltage of repeating unit (RU) *i* in the stack	$V_{RU,i}$	V	V	-
Maximum RU voltage	$V_{RU,max}$	V	V	-
Minimum RU voltage	$V_{RU,min}$	V	V	-
Voltage of the stack	V_{stack}	V	V	-
Thermoneutral voltage	V_{tn}	V	V	-
Angular frequency	ω	rad s^{-1}	rad s^{-1}	1 Hz = 2π rad 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 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	$x_{i,pos,in}$	-	-	-
Mole fraction of component *i* in the positive electrode gas stream at cell/stack outlet	$x_{i,pos,out}$	-	-	-
Number of exchanged electrons	z	-	-	-

Impedance	Z	Ω	Ω	-
Modulus of impedance	$\|Z\|$	Ω	Ω	-
Real part of impedance	Z'	Ω	Ω	-
Imaginary part of impedance	Z''	Ω	Ω	-

Notes:

1. About the unit of gas flow rate. Based on the unit definition of the most commonly used mass flow controllers and meters, the following terms apply: nlpm (normal litre per minute) refers to the volumetric flow rate of gases under normal conditions (0 °C or 273.15 K and 1.01325 bar). **slpm** (**standard litre per minute**) refers to the volumetric flow rate of gases under standard conditions (20 °C or 293.15 K and 1.01325 bar). Mixing up these two reference conditions will cause an error of 7% (293.15/273.15=1.07). Therefore, it is strongly recommended to check the specifications of the mass flow controllers and meters used in test stations before setting the flow rates of gases. In addition, the gas flow rate can be normalized by the active area of the cell or stack. In this case the unit of volumetric gas flow rate will be l_n min^{-1} cm^{-2} or l_s min^{-1} cm^{-2}.

2. To express the rate of change of a certain parameter, a dot above the parameter symbol can be used. For instance $\dot{T}$ can be used to express the temperature ramp with a unit of K min^{-1}, $\dot{f}$ can be used to express the speed of variation of the gas flow rate with a unit of nlpm min^{-1}. Alternatively, ($\Delta X/\Delta t$) can be used. For instance, the temperature ramp can be expressed by ($\Delta T/\Delta t$) and the speed of current density variation can be expressed by ($\Delta j/\Delta t$).

3. About the amplitude of alternating current/voltage with a sine waveform for EIS measurement: it refers here to the peak amplitude. It amounts to half of the *peak-to-peak amplitude* and $\sqrt{2}$ times the *root mean square (RMS) amplitude*.

4. The real part and imaginary part of impedance can also be represented by *Re(Z)* and *Im(Z)*, respectively.

8 Definition of test objective, test program and test module

The SOCTESQA project defines test programs that have different objectives and scopes. The test programs are comprised of a series of test modules (Figure 10), in which the actual test parameters, specific conditions and test setup are described in detail. The test program describes the objective and scope of the test as well as the test operating conditions (TOC), where the general application specific parameters are given, such as reactant composition, operating temperature etc.

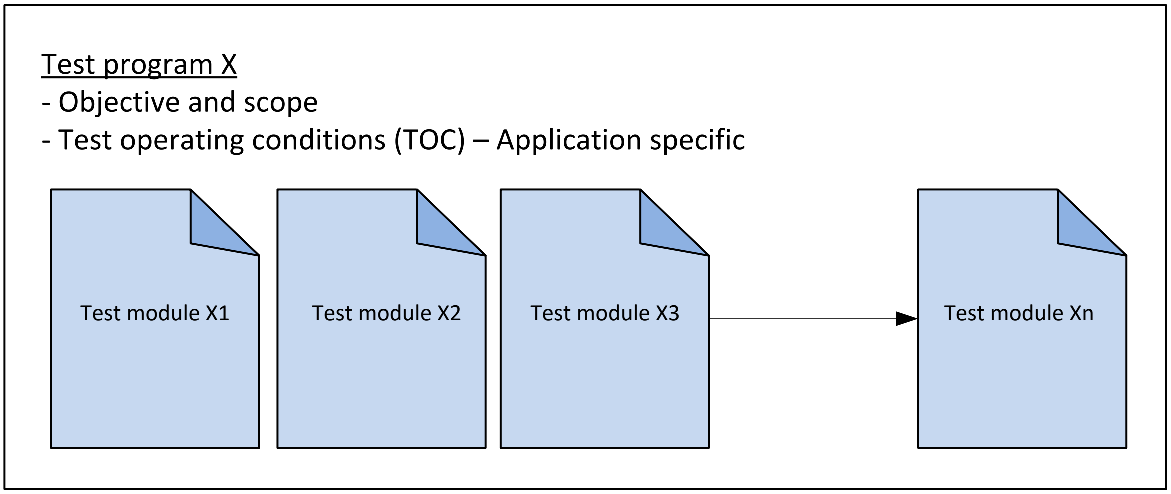

Figure 10: Scheme of a test program consisting of a series of test modules

More than one test program can be run in sequence, whereby time and thermal stress from start-up and shut-down in test programs can be minimized.

SOCTESQA will suggest several test programs, but there is no limitation for others to create their own test programs as needed. Nevertheless, we suggest the following regarding the design of test programs, as it allows for better comparison and benchmarking of results, if this is desired.

- Most test programs will comprise a start-up and a shutdown module to safely start up the stack and document the initial and final performance of the SOC cell/stack.
- Each of these test programs may contain several test modules for characterization, e.g. current-voltage characteristics, electrochemical impedance spectroscopy (EIS) and performance and durability testing, with e.g. dynamic operating profiles or long term stationary operation.

The SOCTESQA test programs will address three different operation modes, solid oxide fuel cell (SOFC), solid oxide electrolysis cell (SOEC) and combined SOFC/SOEC operations. Each of these cases includes both performance and durability. This will cover many potential application requirements, e.g. micro-combined heat and power generation (micro-CHP), auxiliary power unit (APU), SOEC for H_2 production (Power-to-gas) or combined SOFC/SOEC energy conversion systems (Power-to-gas-to-power).

8.1 Test objective and scope

The determination of the objective of a test is the initial part of planning the procedure for testing a ceramic solid oxide cell/stack. The objectives could be:

- Evaluation of the performance
- Stability over time at constant conditions
- …

With the objective of the test in mind, the test can be planned by first choosing the relevant scope, that covers the relevant test operating condition (depending on type of application), operating mode, cell, short-stack or stack testing. Figure 11 shows an example. SOCTESQA defines a set of recommended values for test operating conditions for different applications.

Test program: Temperature

Objective and scope: To investigate the influence of the cell/stack temperature on performance, in terms of cell/stack voltage (SOFC), or hydrogen production (SOEC).

It can be used to determine the sensitivity of an SOC cell/stack to the nominal operating temperature as well as to the variation in the cell/stack temperature at varying current.

Figure11: Example of test objective and scope

8.2 Test program

Test programs comprise a set of test modules, each module with its own specific objective, as well as the test program objective. The test program specifies the order of test modules, the number and in some instances also parameters such as total test time, e.g. in the case of durability testing where you would need intermittent characterisation throughout the test. Figure 12 shows an example of this.

Test programs can ideally be planned in sequence to investigate several objectives, and thus the shutdown and start-up in between can be omitted.

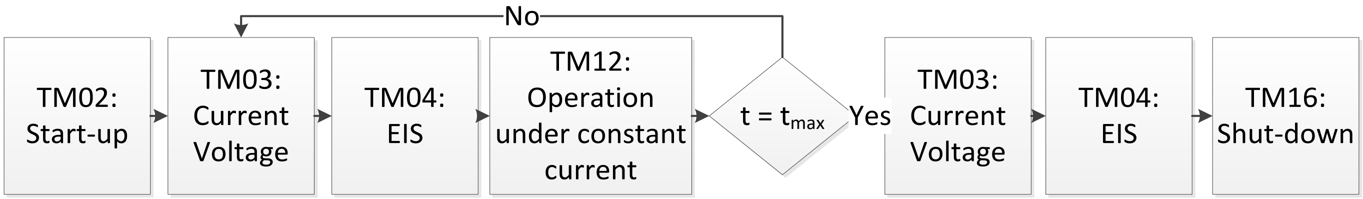

Figure 12: Example of a test program structure, comprising several test modules for characterization and performance evaluation

8.3 Test module

A test module is a confined test procedure in which a minimum of test input parameters is changed to achieve a desired effect. The test module is carried out under the general test operating conditions set in the test program, unless stated in the module.

The test module describes input and output parameters to be controlled and/or measured.

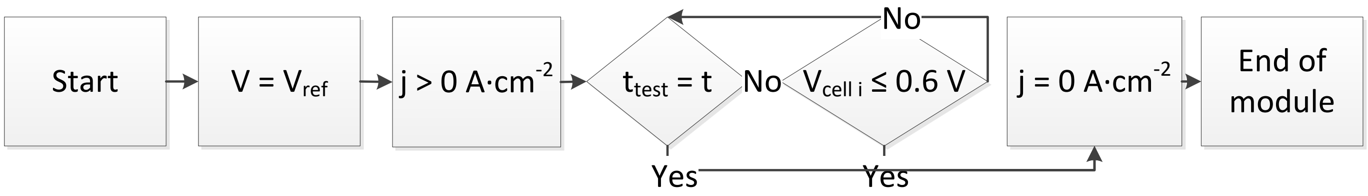

Figure 13: Example of a test module, for durability testing under constant current in SOFC operation

9 Reporting of testing results

The report of the test results should contain all necessary information for the evaluation of the test object. The following templates and tables contain recommendations for a generic test report on SOC test objects, including single cells or stacks under SOFC, SOEC or combined SOFC/SOEC operation.

9.1 Objective and scope of the test

Here the authors should present the objective and the scope of the test.

9.2 Introduction and test planning

Here the author should refer to:

- The Test Modules used to formulate the testing procedure.
- The procedure applied, and if relevant, explain the choice of this procedure.
- The test plan between tester and customer which may also include acceptance criteria and any other documentation used in the report or in the test (terminology document, symbols harmonization etc.).

9.3 General information concerning the test

Table 5: General information on the test report

Test report reference/identification	
Test report title	
Authors	
Date of report	

Table 6: General information concerning the test

Test module / test program number	
Test version	
Company requesting test	
Test Request No	
Person performing the test	

Test date	
Company performing test	
Test location	
Test stack	
Test station ID	

Table 7: Test object description

Test object manufacturer	
Operational mode (SOFC, SOEC or combined)	
Test object model	
Number of cells N	
Product or object tested	
Product number	

Test object identity number	
Test object dimensions (H x L x W) (cm^3)	
Test object weight (kg)	
Test object nominal power (W)	
Test object peak power (W)	
Test object minimum power (W)	
Test object voltage range (V)	
Lowest/highest voltage allowed (V)	
Lowest/highest allowed individual single cell voltage (mV)	
Maximum allowed pressure difference between anode and cathode (kPa)	
Maximum allowed temperature difference between gas inlet and outlet temperature (°C)	

Solid oxide cell (SOC): material/coating of the bipolar plates / technology	
Solid oxide cell (SOC): flow field design	
Solid oxide cell (SOC): active area (cm^2)	
Gasket type	
Gasket thickness	
Minimum air flow rate (nlpm)	
Compression force (N)	

Negative electrode material	
Positive electrode material	
Electrolyte material	
Negative electrode thickness	
Positive electrode thickness	
Electrolyte thickness	

Test object leakage $ml_n cm^{-1} min^{-1}$	

The author has to precise the origin of the data (manufacturer or in-house). Proprietary information labelled as such whenever a reference of such data is described in the IEC 62282-2 Part 2: Fuel Cell Module, standard.

9.4 Status of the test object

The author presents here the testing history of the test object with a short description of all diagnostic experiments, specific or baseline experiments and their respective identifiers in sequential order. All test modules and test programs already realized should be mentioned.

9.5 Description of the test setup

A detailed description of the used test equipment and setup, including sensors type and location and specific devices (for example, heating/cooling and humidification sub-systems), has to be presented to aid understanding the test results. A graphical representation may be added here.

Table 8: List of sensors for test setup

Sensor name	**Sensor type**	**Sensor location**
…	…	…

9.6 Description of the operating conditions, TIPs and TOPs

Here the following shall be presented:

- The settings of the operating conditions (TIPs).
- The cause of ending the test (where applicable).
- Measurement method(s) of the test module(s) and any deviation from it.

Table 9: Test Input Parameters (TIPs)

TIP	**Descrip-tion**	**Minimum value (unit of TIP)**	**Maximum value (unit of TIP)**	**Standard deviation (unit of TIP)**	**Relative standard deviation (unit of TIP)**	**Standard error (unit of TIP)**	**Sampling rate (Hz)**
TIP$_1$							
...							
TIP$_n$							

Table 10: Test Output Parameters (TOPs)

TOP	**Descrip-tion**	**Minimum value (unit of TOP)**	**Maximum value (unit of TOP)**	**Standard deviation (unit of TOP)**	**Relative standard deviation (unit of TOP)**	**Standard error (unit of TOP)**	**Sampling rate (Hz)**
TOP$_1$							
...							
TOP$_n$							

Graphs may be added here to present the main test inputs and outputs, represented as described in the individual Test Modules used.

Plots of TIPs, TOPs and derived quantities may be grouped in four categories:

- Performance diagrams or maps,
- Stability diagrams
- Property diagrams
- Homogeneity diagrams

The plots that may be included in each of these categories may vary according to the operational mode (SOFC, SOEC or combined) of the test object.

Graphs of the main test inputs and test outputs of the stack versus dwell time or test duration during start-up and conditioning may be included here (and where appropriate for individual cells), to aid the understanding of the stability of such parameters for these test steps.

Performance diagrams / maps

A performance map may include TOPs or derived quantities plotted against TIPs or derived quantities that are directly related to the performance of the test object. Possible plots that may be included in this category are:

- Voltage or power density versus current density, fuel utilization, gas composition, inlet pressure or temperature
- Efficiency versus temperature, inlet pressure, fuel utilization or gas composition
- Hydrogen/oxygen production versus temperature, steam content or pressure
- ….

Stability diagrams

- Degradation or its rate, load cycling, impedance plots versus temperature, test duration, inlet pressure, fuel utilization or gas composition
- …….

Property diagrams

Property diagrams may include plots of derived quantities, reflecting the properties of the test object. Possible plots that may fall into this category are:

- Power density versus Area Specific Resistance, polarization resistance, ohmic resistance, or efficiency
- ….

Homogeneity diagrams

Homogeneity diagrams may include plots of TOPs against local test specimen property:

- Power density versus stack layer
- Current density versus x-position of cell area
- Temperature versus height of stack assembly unit
- gas flow rate against width of cell
- ….
- ….

9.7 Data (Post) Processing

Here, the post processing of the test results (TIPs and TOPs) shall be described.

When reporting, the significant figures of a test variable should be consistent with its measurement uncertainty. For a calculated test variable, the lesser number of significant figures of all of the involved test variables should determine the significant figure. The standard deviation (stdev), relative standard deviation (RSD) and standard error (stderr) should be expressed with at least one additional figure. The RSD should be reported with two significant figures.

10 Appendix (constants and formulary)

<table>
<tr><th>Description</th><th>Constants and Formulary</th></tr>
<tr><td>Faraday constant</td><td>F = 96485.3 C mol^{-1} = 96485.3 A s mol^{-1}</td></tr>
<tr><td>Oxygen fraction in air</td><td>x_{O2} = 0.2095</td></tr>
<tr><td>Lower heating value (LHV) of H_2[7]</td><td>LHV_{H2} = 119.93 kJ g^{-}1 = 241.77 kJ mol^{-1}</td></tr>
<tr><td>Higher heating value (HHV) of H_2[7]</td><td>HHV_{H2} = 141.86 kJ g^{-1} = 285.98 kJ mol^{-1}</td></tr>
<tr><td>Voltage equivalent to LHV of H_2</td><td>$LHV_{H2} / (2F) = 1.253\ V$</td></tr>
<tr><td>Voltage equivalent to HHV of H_2</td><td>$HHV_{H2} / (2F) = 1.482\ V$</td></tr>
<tr><td>Molar gas constant</td><td>R = 8.31446 J K^{-1} mol^{-1}</td></tr>
<tr><td>Normal temperature</td><td>T_n = 273.15 K</td></tr>
<tr><td>Normal pressure</td><td>P_n= 101325 N m^{-2} = 101325 Pa</td></tr>
<tr><td>Molar volume of an ideal gas at normal temperature and pressure</td><td>$V_m = RT_n/P_n$ = 8.31446 x 273.15 / 101325 m_n^3 mol^{-1} = 22.414 l_n mol^{-1}</td></tr>
<tr><td>Number of electrons transferred when one molecule of reactant component i is electrochemically reacted (z_i)</td><td>
<table>
<tr><th>i</th><th>z_i</th><th>*Electrochemical reaction*</th></tr>
<tr><td>H_2</td><td>2</td><td>$H_2 + O^{2-} \rightarrow H_2O + 2e^-$</td></tr>
<tr><td>CO</td><td>2</td><td>$CO + O^{2-} \rightarrow CO_2 + 2e^-$</td></tr>
<tr><td>CH_4</td><td>8</td><td>$CH_4 + 4O^{2-} \rightarrow CO_2 + 2H_2O + 8e^-$</td></tr>
<tr><td>$C_pH_qO_r$</td><td>4p+q-2r</td><td>$C_pH_qO_r + (2p+q/2-r)O^{2-} \rightarrow pCO_2 + q/2H_2O + (4p+q-2r)e^-$</td></tr>
<tr><td>O_2</td><td>4</td><td>$O_2 + 4e^- \rightarrow 2O^{2-}$</td></tr>
<tr><td>H_2O</td><td>2</td><td>$H_2O + 2e^- \rightarrow H_2 + O^{2-}$</td></tr>
</table>
</td></tr>
<tr><td>Gas utilization (U_{gas})</td><td>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):</td></tr>
</table>

	$I_{theory} = \frac{F}{V_m \times 60} \cdot \frac{\sum_{i=1}^{n} z_i \times f_{i,in}}{N} = \frac{96485.3}{22.414 \times 60} \times \frac{\sum_{i=1}^{n} z_i \times f_{i,in}}{N}$ $= 71.74 \times \frac{\sum_{i=1}^{n} z_i \times f_{i,in}}{N}$ gas utilization at current *I*: $U_{gas} = \frac{I}{I_{theory}} \times 100\% = \frac{I \times N}{71.74 \times \sum_{i=1}^{n} z_i \times f_{i,in}} \times 100\%$
Nernst voltage / reversible (thermodynamic) voltage / theoretical open circuit voltage (V_N)	$V_N = \frac{RT}{4F} ln \frac{p_{O2,pos}}{p_{O2,neg}}$ (considering SOC as an oxygen concentration cell) $V_N = \lvert \frac{\Delta^r G(T,p)}{zF} \rvert$ (for any reaction) $\Delta^r G(T,p)$: Gibbs free enthalpy of reaction as a function of temperature and pressure. For the reaction: H_2 + 0.5 O_2 = H_2O $\Delta^r G(T,p) = \Delta^r G^0(T) - RT ln \frac{p_{O2,pos}^{\frac{1}{2}} p_{H2,neg}}{p_{H2O,neg}}$ $V_N = -\frac{\Delta^r G(T,p)}{2F} = -\frac{\Delta^r G^0(T)}{2F} + \frac{RT}{2F} ln \frac{p_{O2,pos}^{\frac{1}{2}} p_{H2,neg}}{p_{H2O,neg}}$ $= V_N^0(T) + \frac{RT}{2F} ln \frac{p_{O2,pos}^{\frac{1}{2}} p_{H2,neg}}{p_{H2O,neg}}$ $\Delta^r G^0(T)$: Gibbs free enthalpy of reaction at standard pressure $V_N^0(T)$: reversible voltage at standard pressure
Thermoneutral voltage (V_{tn})	$V_{tn} = \frac{\Delta^r H(T)}{zF}$ $\Delta^r H(T)$: Enthalpy of reaction as a function of temperature. (Note: the enthalpy is independent of the pressure under the assumption of ideal gases). *z*: number of exchanged electrons in the electrochemical reaction. For water electrolysis reaction: H_2O → H_2 + 0.5 O_2 V_{tn} = 1.482 V at 20 °C, 1.283 V at 700 °C, 1.285 V at 750 °C and 1.286 V at 800 °C
Average RU voltage ($V_{RU,av}$)	$V_{RU,av} = \frac{\sum_{i=1}^{N} V_{RU,i}}{N}$
Electrical power of the cell / stack (P_{el})	$P_{el} = V_{cell/stack} \times I$
(Area specific)	$P_{d,el} = \frac{P_{el}}{A \times N}$

electrical power density	
Electrical efficiency of the stack (SOFC mode)	The electrical efficiency of an SOFC stack can be defined as the ratio of electric power output to the total enthalpy flow input (based on either LHV of HHV of feed fuel gases). $$\eta_{el,LHV} = 22.414 \times 60 \times \frac{P_{el}}{\sum_{i=1}^{n} LHV_i \times f_{i,neg,in}}$$ $$\eta_{el,HHV} = 22.414 \times 60 \times \frac{P_{el}}{\sum_{i=1}^{n} HHV_i \times f_{i,neg,in}}$$ LHV_i: LHV of fuel component *i* (J mol^{-1}) HHV_i: HHV of fuel component *i* (J mol^{-1}) $f_{i,neg,in}$: flow rate of fuel component *i* (*i* = 1 … *n*)(nlpm) When using H_2 as fuel: $$\eta_{el,LHV} = \frac{U_{gas,neg} \times V_{stack}}{1.253 \times N}$$ $$\eta_{el,HHV} = \frac{U_{gas,neg} \times V_{stack}}{1.482 \times N}$$
Electrical efficiency of the stack (SOEC mode: H_2O electrolysis)	The electrical efficiency of an SOEC stack can be defined as the ratio of enthalpy flow of fuel gases produced by the electrolyzer (based on either LHV of HHV of produced fuel gases) to the electrical power consumed by the stack for the electrochemical reaction. Here electrical power consumed by the water evaporator, gas preheaters and the furnace in the test station is not considered. It should be noted that for the calculation of system efficiency, these consumptions have to be taken into account. For H_2O electrolysis and assume 100% current efficiency: $$\eta_{el,LHV,H2-production} = \frac{1.253 \times N}{V_{stack}}$$ $$\eta_{el,HHV,H2-production} = \frac{1.482 \times N}{V_{stack}}$$
Degradation	The absolute degradation ΔX of a quantity X within the time from t_0 to t_1 is calculated as the difference between the final value $X(t_1)$ and the initial value $X(t_0)$: $$\Delta X = X(t_1) - X(t_0)$$ The relative degradation ΔX_{rel} is calculated by dividing ΔX by the initial value $X(t_0)$: $$\Delta X_{rel} = \frac{X(t_1) - X(t_0)}{X(t_0)} \times 100\%$$

	The degradation rate (rate of change) of quantity X during the time interval (t_1-t_0) is then calculated by: $\frac{\Delta X}{\Delta t} = \frac{\Delta X}{t_1 - t_0}$ (with the unit [unit of X/time unit]) $\frac{\Delta X_{rel}}{\Delta t} = \frac{\Delta X_{rel}}{t_1 - t_0}$ (with the unit [%/time unit]) Degradation rates are typically expressed by the absolute or relative change per 1000 hours. It is thus advisable to normalize the results to 1000 h time interval. This can be simply done by converting the unit of time interval to kh. Example: An SOFC stack with 5 RUs shows a stack voltage of 4.500 V at t_0 = 500 h. At t_1=1300 h, the stack voltage dropped to 4.482 V. The absolute and relative degradation rates of the stack voltage during time interval 500-1300 h are: $$\frac{\Delta V_{stack}}{\Delta t} = \frac{V_{stack}\,(t_1) - V_{stack}\,(t_0)}{t_1 - t_0} = \frac{V_{stack}\,(1300\,h) - V_{stack}\,(500\,h)}{(1300-500)h} = \frac{(4.482-4.500)\,V}{(1300-500)\,h} = \frac{-0.018\,V}{800\,h} = \frac{-0.018\,V}{0.8\,kh} = -0.0225\,V\,kh^{-1}$$ $$\frac{\Delta V_{stack,rel}}{\Delta t} = \frac{V_{stack}\,(t_1) - V_{stack}\,(t_0)}{V_{stack}\,(t_0) \times\ (t_1 - t_0)} \times 100\% = \frac{V_{stack}\,(1300\,h) - V_{stack}\,(500\,h)}{V_{stack}\,(500\,h) \times (1300-500)h} \times 100\% = \frac{4.482-4.500}{4.500\ \times (1300-500)\,h} \times 100\% = \frac{-0.4\%}{800\,h} = \frac{-0.4\%}{0.8\,kh} = -0.5\%\,kh^{-1}$$ Additionally, when long-term cycling is performed (thermal or load cycles for instance), it is common and relevant to express the degradation rate in relation to the number of cycles *m* as follows for absolute and relative degradation: $$\Delta X_m = \frac{X(t_1) - X(t_0)}{m}$$ $$\Delta X_{m,rel} = \frac{X(t_1) - X(t_0)}{X(t_0) \cdot m} \cdot 100\%$$
Area specific resistance (ASR)	The area specific resistance can be determined from the *j*-*V* characteristic. Therefore, a small voltage interval where the current voltage curve is nearly linear is needed. The difference in voltage ($\Delta V(j)$) divided by the difference of the corresponding current density (Δj) is used to calculate the ASR.

	$ASR(j) = \lvert \frac{\Delta V(j)}{\Delta j} \rvert$ Note that the ASR is dependent on the current/current density. In the non-linear region of the *j*-*V* curve, it is recommended to choose small voltage and current intervals.
Temperatures	Some stack designs do not allow 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 exemplarily as follows: $T_{av} = \frac{T_{TP}+T_{BP}+T_{neg,in}+T_{neg,out}+T_{pos,in}+T_{pos,out}}{6}$ A stack can be damaged during the start-up/shut-down 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/shut-down can be calculated with the following formula if the internal temperature cannot be measured directly: $\Delta T_{max} = \left\lvert \frac{(T_{neg,in}+T_{pos,in})}{2} - \frac{(T_{TP}+T_{BP})}{2} \right\rvert$
Electrochemical Impedance Spectroscopy	Alternating current signal (galvanostatic mode) in the time domain: $I(\omega, t) = \bar{I} \sin(\omega t)$ Angular perturbation frequency: $\omega = 2\pi\upsilon$ Alternating voltage response in the time domain: $V(\omega, t) = \bar{V} \sin(\omega t + \varphi)$ Impedance *Z(ω)* of an electrochemical component in the time domain: $Z(\omega) = \frac{V(\omega, t)}{I(\omega, t)} = \frac{\bar{V} \sin(\omega t + \varphi)}{\bar{I} \sin(\omega t)} = \lvert Z \rvert \cdot \frac{\sin(\omega t + \varphi)}{\sin(\omega t)}$ Impedance in the frequency domain (Fourier transform, FT space): $Z(\omega) = \frac{FFT\{V(\omega,t)\}}{FFT\{I(\omega,t)\}} = \lvert Z \rvert\, exp(i\varphi) = \lvert Z \rvert \cos(\varphi) + \lvert Z \rvert i \sin(\varphi) = Z + i \cdot Z''$, Magnitude or modulus of the impedance: $\lvert Z(\omega) \rvert = \sqrt{Z'(\omega)^2 + Z''(\omega)^2}$ $tan\varphi(\omega) = \frac{Z''(\omega)}{Z'(\omega)}$

	Imaginary unit property: $i^2=-1$

11 Bibliography

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6. EU-Project Stacktest: Stack-Test Master Document – TM2.00,http://stacktest.zsw-bw.de/fileadmin/stacktest/docs/Information_Material/Performance/TMs/TM_P-00_Stack-Test_Master-Document.pdf

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Test Module 02:
Start-up

(Version 1.16, 14 March 2017)

Objective and Scope

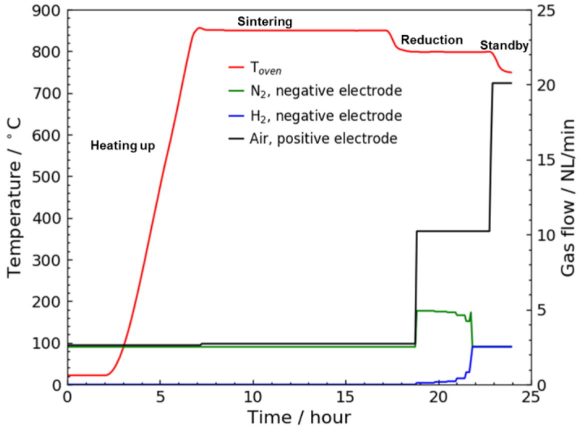
This test module deals with the start-up procedure of solid oxide cells/stacks needed to achieve operating conditions. Start-up includes the heating step and if needed, the gas tightness and electrical contact optimization, the reduction and the conditioning of the SOC stack or cell. The start-up procedure should be given by the manufacturer. However, if there is no start-up procedure available, a recommendation is given in this test module.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Rate of oven temperature change ($\Delta T_{oven}/\Delta t$)	Temperature of the oven (T_{oven})
	Temperature of the pre-heater ($T_{PH,in}$)
	Flow rates of inlet gases (f_{in})
	Composition of inlet gases ($x_{i,\ in}$)

Test Procedure

- Set the flow for the negative ($f_{neg,in}$) electrode to N_2 or Ar. Set the flow for the positive electrode ($f_{pos,in}$) to air.
- Increase cell/stack temperature by setting T_{oven} to the required sealing temperature. Increase the gas inlet temperatures (if possible) by adjusting $T_{PH,in}$.
- Hold time at sealing temperature according to setup requirements.
- Change T_{oven} to the reduction temperature required by the SOCs. Initiate the reduction process (e.g. by increasing H_2 flow stepwise).
- Change T_{oven} to the operating temperature; change the reactants to nominal operation flow/composition.



Schematic example of start-up procedure for non-reduced SOC cell/stack

Critical Parameters and Parameter Controls

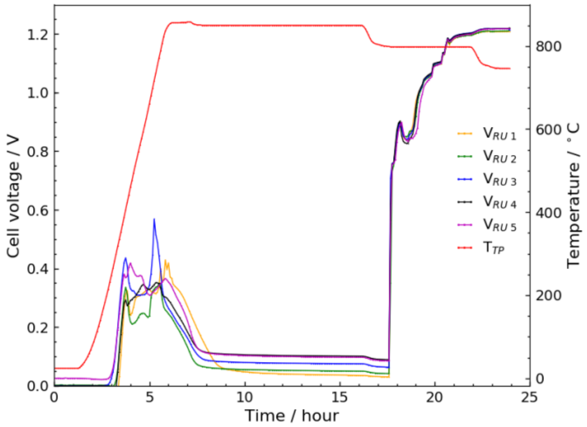
- A large temperature gradient between the gas inlets and the cell/stack should be avoided to reduce the risk of cell/stack damage

Main Test Output Parameters (TOPs) and Derived Quantities

TOP	Derived Quantities
Voltage of cell/stack (V)	Maximum temperature difference during start-up (ΔT_{max})
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	

Data Post Processing and Representation

Representation examples of start-up:



Schematic example of startup procedure for non-reduced 5-cells SOC stack

SOCTESQA:
Solid Oxide Cell and Stack Testing, Safety and Quality Assurance
Project website: www.soctesqa.eu

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Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU GRANT AGREEMENT N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
***Start date**: 01.05.2014 – **Duration**: 36 months*
***Project Coordinator**: M. Lang – DLR*

Test Module 02: Start-up

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Content

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	cm²	m²
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:

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

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:

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

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.

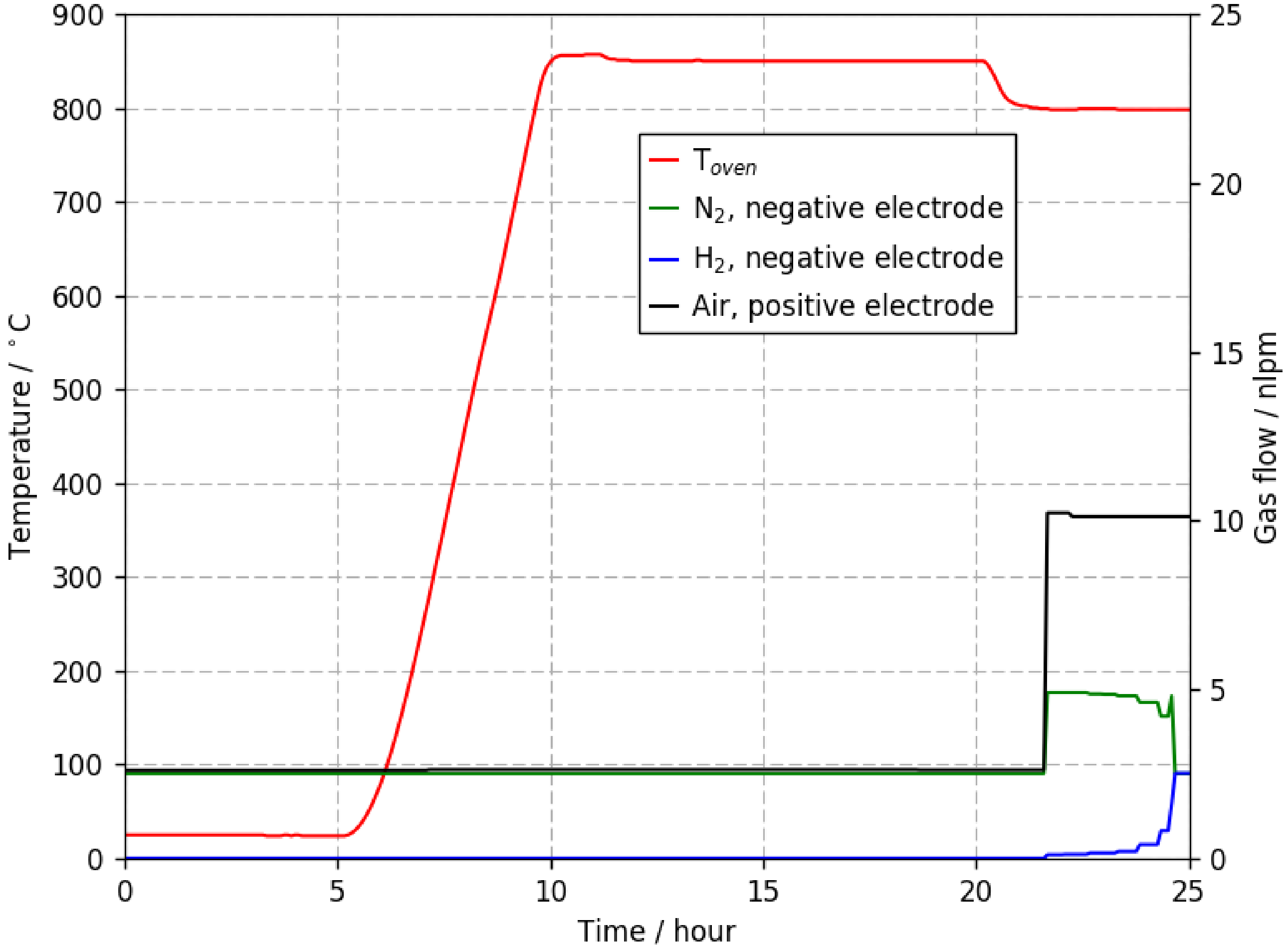

Figure 1 Schematic example of a start-up procedure for non-reduced SOC 5 cells stack

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.

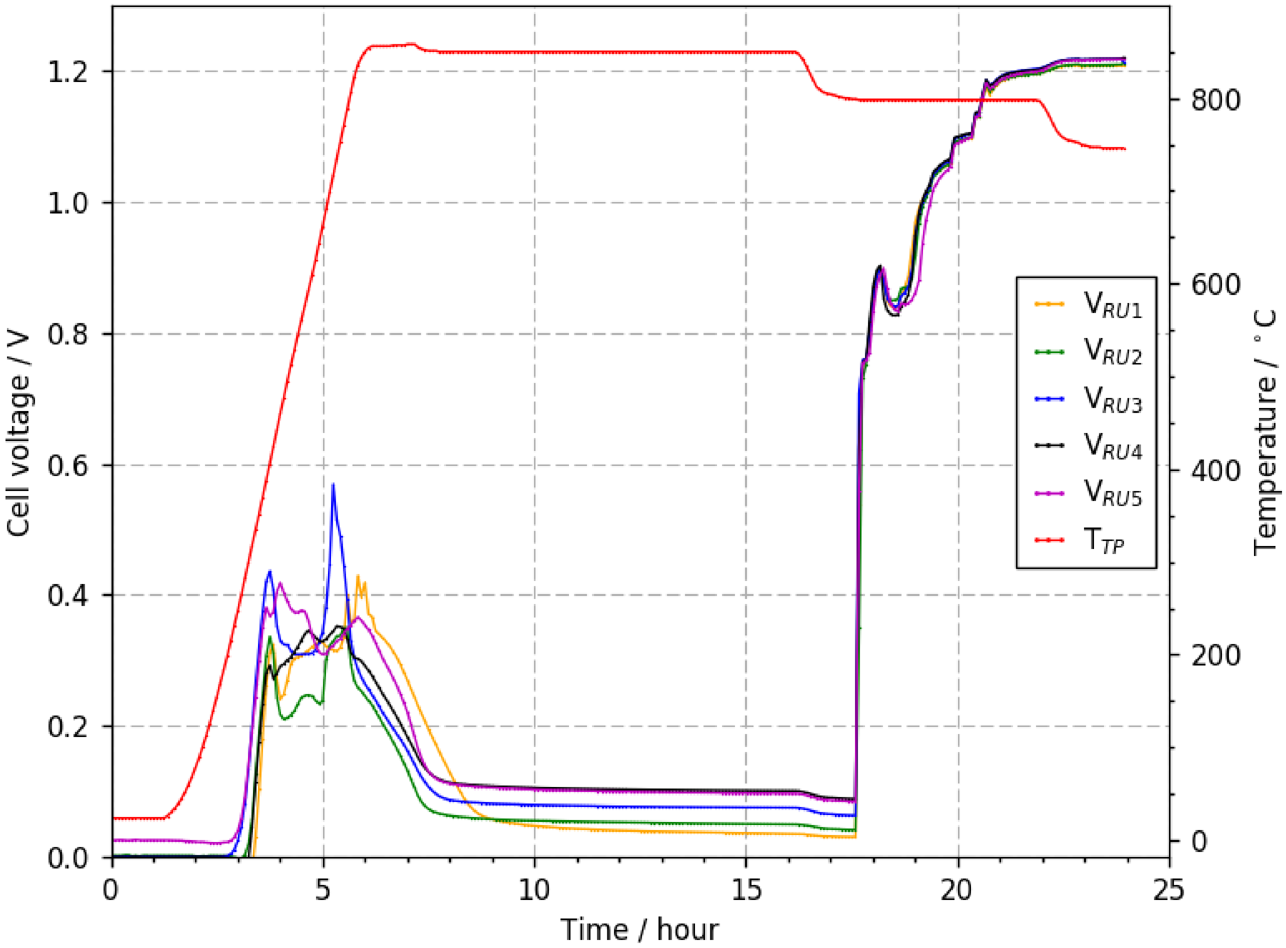

Figure 2*: Voltages and top plate temperature of the repeating units of a 5 cells non-reduced SOC short stack during start-up*

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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2. WP2 Stationary fuel cell systems, Test Programs and Test Modules, FC-TESTNET project, 2006.
3. SOFC single cell performance and endurance test modules, Test Modules TM SOFC 01-04LD / 05-08HD, FCTESQA project, 2010.
4. SOFC stacks performance and endurance test modules, Test Modules TM SOFC 25-28HD, FCTESQA project, 2010.

Test Module 03
Current-voltage Characteristics

(Version 1.19, 15 May 2017)

Objective and Scope

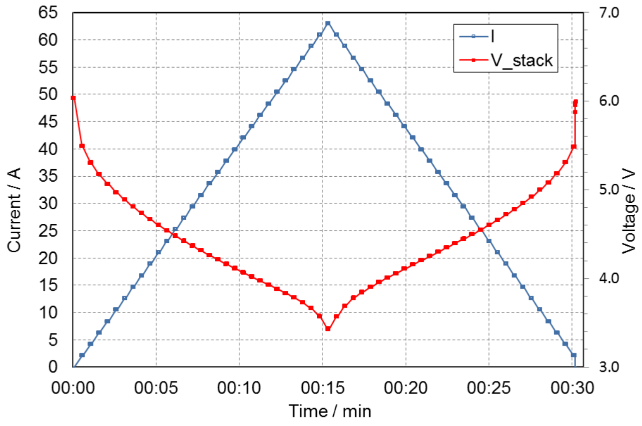
This test module deals with solid oxide cell (SOC) operation at different current densities either as a fuel cell (SOFC) or as an electrolyser (SOEC) to determine the current-voltage characteristics (*j*-*V*, polarization curve measurement) of an SOC cell/stack. It is a general characterization method that can be used in SOC R&D and for quality assurance.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Rate of change of current ($\Delta I/\Delta t$)	Current (I)
Flow rates of inlet gases (f_{in})	
Temperature of the oven (T_{oven})	
Pressure of outlet gases (p_{out})	
Composition of inlet gases ($x_{i,\ in}$)	

Test Procedure

- Stepwise increase current from zero (open circuit voltage) to current at the specified cut-off voltage (ascending *j*-*V* curve) followed by current decrease to zero (descending *j*-*V* curve) using the specified rate of current change, e.g. 1 mA cm^{-2} sec^{-1}.
- Continuously record all TIPs & TOPs at their specified sampling rates, e.g. 1 Hz.
- For step duration of longer than 1 second, the values of SOC voltage last measured during each step versus the current density constitute the data points of the *j*-*V* curve.



Evolution of SOFC stack current and voltage during test (ascending and descending)

Critical Parameters and Parameter Controls

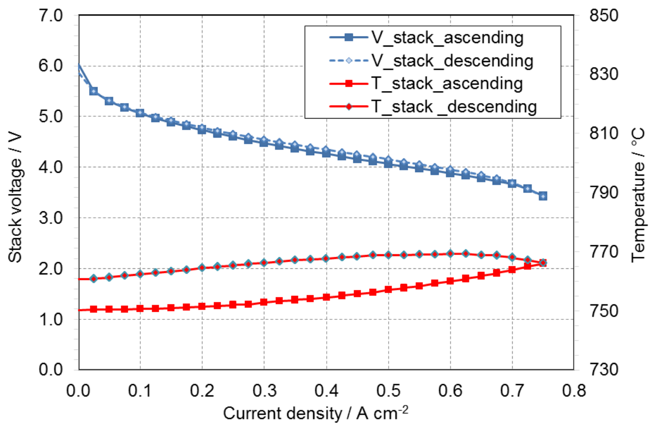
- Stability of T_{cell}, T_{stack}, $T_{neg,in}$, $T_{pos,in}$, $T_{neg,out}$ and $T_{pos,out}$ under OCV conditions (zero current) prior to the actual polarization (*j*-*V*) curve measurement (current-voltage characteristics).
- In SOEC mode, special attention is to be paid to a stable supply of steam to limit SOC voltage fluctuations to within a specified value at OCV, e.g., ±10 mV per cell.

Main Test Output Parameters (TOPs) and Derived Quantities

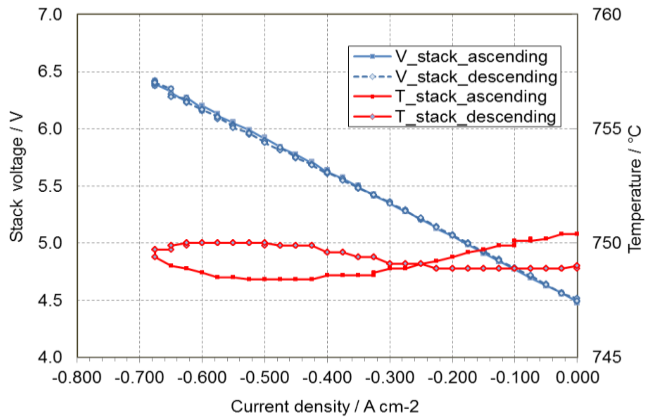
TOP	Derived Quantity
Voltage of cell/RU/stack (V)	Current density (j)
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	Electrical power density ($P_{d,el}$)
	Reactant gas utilization (U_{gas})
	Area specific resistance (ASR)

Data Post Processing and Representation

Representation examples of *j*-*V* curves:



SOFC stack *j*-*V* curve (ascending and descending)



SOEC stack *j*-*V* curve (ascending and descending)

SOCTESQA:
Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Project website: www.soctesqa.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU Grant Agreement N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
***Start date**: 01.05.2014 – **Duration**: 36 months*
***Project Coordinator**: M. Lang – DLR*

Test Module 03: Current-voltage Characteristics

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Content

Abbreviations

ASR	Area specific resistance
HHV	Higher heating value
LHV	Lower heating value
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 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* 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 $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
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* in the negative electrode gas stream at cell/stack inlet	$x_{i,neg,in}$	-	-
Mole fraction of component *i* in the negative 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	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	$\Omega\ cm^2$	$\Omega\ m^2$
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 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}
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

$$P_{d,el} = \frac{P_{el}}{A \times N} \tag{4}$$

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

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

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:

$$V = a + b \cdot j + c \cdot j^2 + d \cdot j^3 + e \cdot j^4 + f \cdot j^5 + \cdots \tag{6}$$

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 *ASR*s 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

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

and

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

For hydrogen, for example, the HHV and LHV are 285.98 kJ mol^{-1} and 241.77 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

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

and

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

respectively.

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

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

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}, $V_{\mathrm{RU,i}}$) 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 ($V_{\mathrm{RU,i}}$) 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}, $T_{\mathrm{neg,in}}$, $T_{\mathrm{pos,in}}$, $T_{\mathrm{neg,out}}$ and $T_{\mathrm{pos,out}}$ 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.

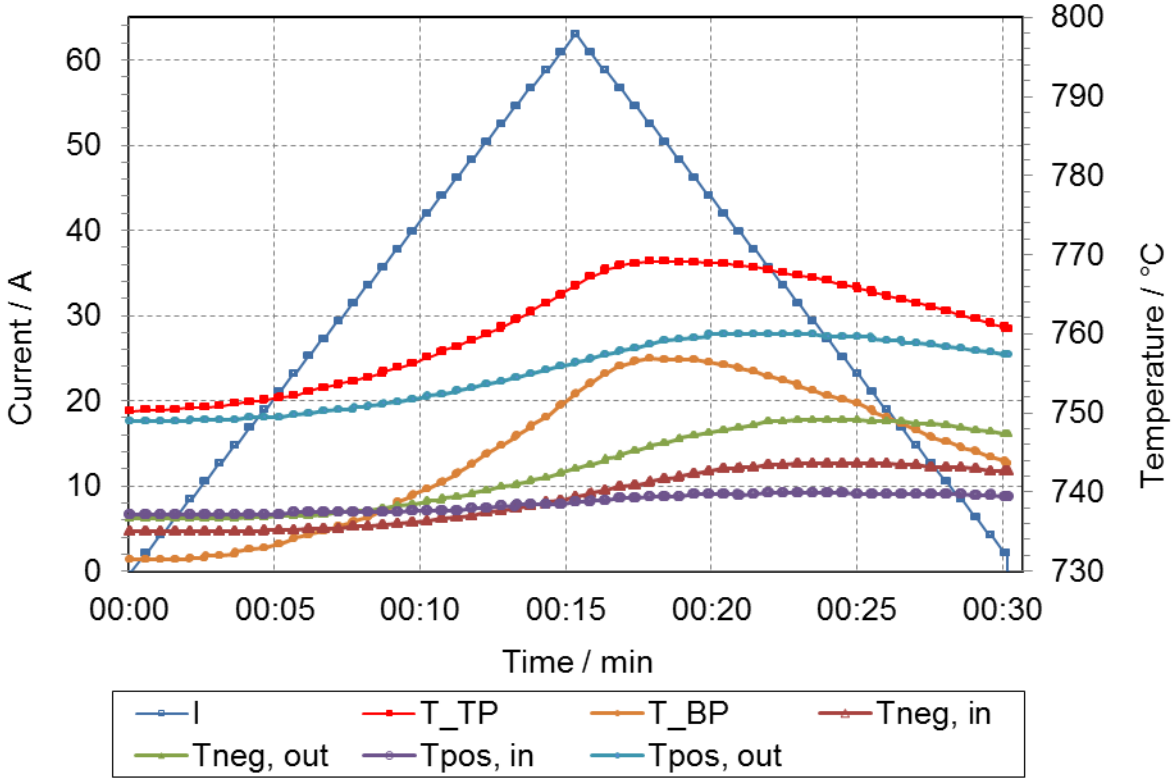


Figure 1: Example of TIP (current) and TOPs (SOFC temperatures) recorded during j-V curve measurement (ascending and descending) showing their evolution.

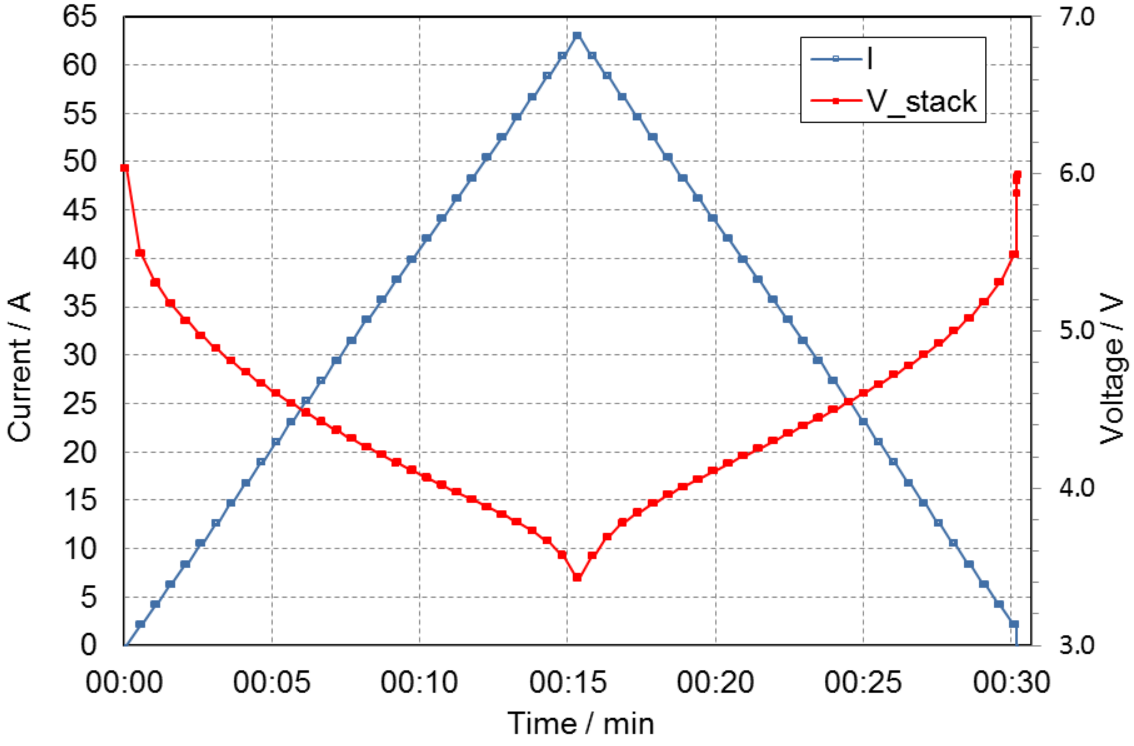


Figure 2: Example of TIP (current) and TOPs (SOFC stack voltage) recorded during j-V curve measurement (ascending and descending) showing their evolution.

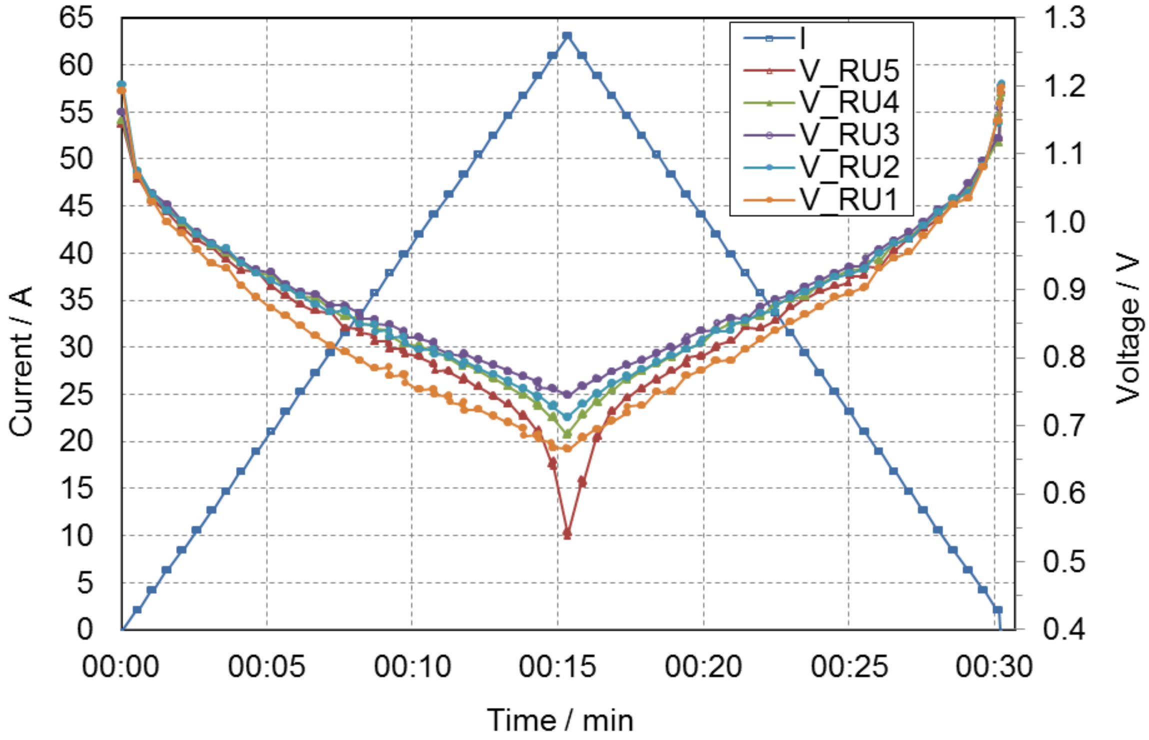


Figure 3: Example of TIP (current) and TOPs (SOFC RU voltages) recorded during j-V curve measurement (ascending and descending) showing their evolution.

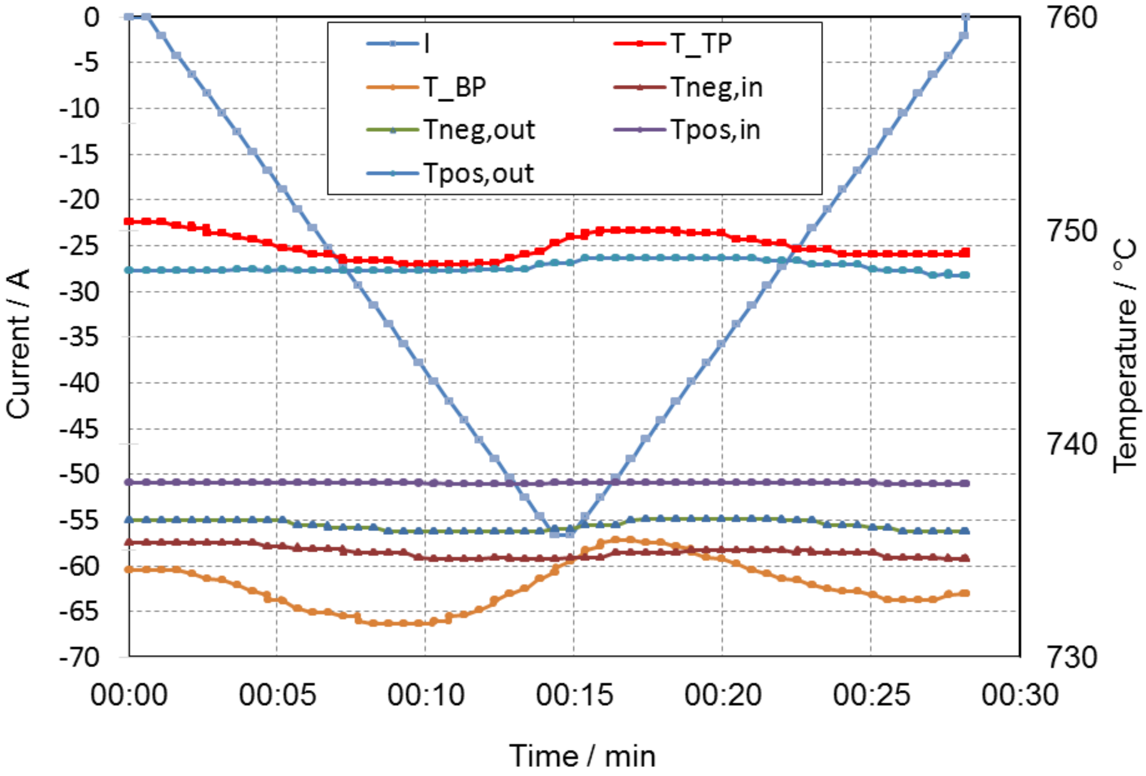


Figure 4: Example of TIP (current) and TOPs (SOEC temperatures) recorded during j-V curve measurement (ascending and descending) showing their evolution.

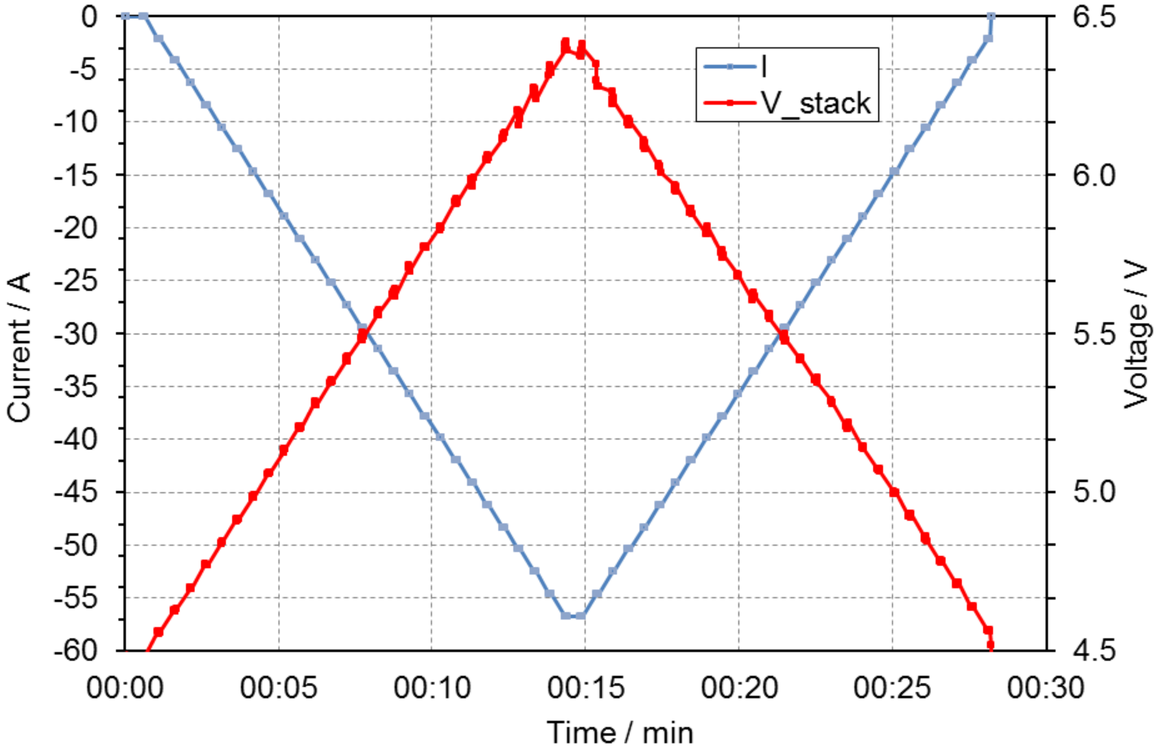


Figure 5: Example of TIP (current) and TOPs (SOEC stack voltage) recorded during j-V curve measurement (ascending and descending) showing their evolution.

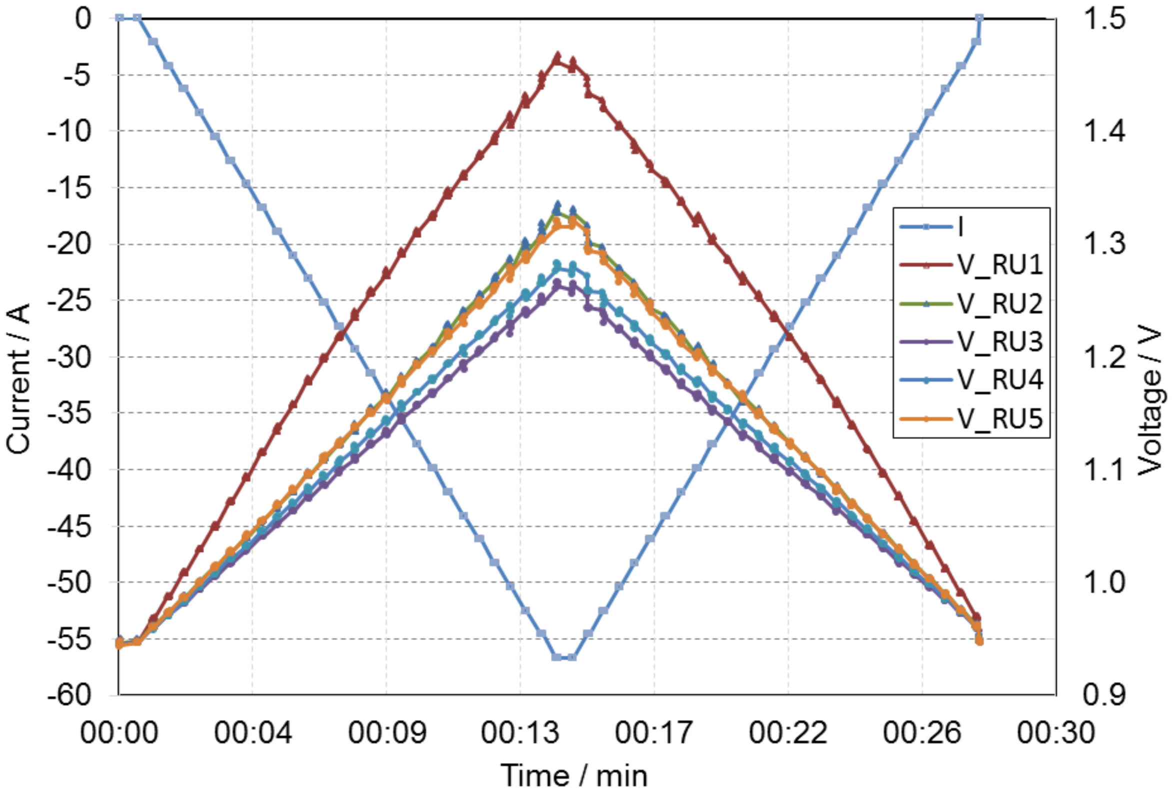


Figure 6: Example of TIP (current) and TOPs (SOEC RU voltages) recorded during j-V curve measurement (ascending and descending) showing their evolution.

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.

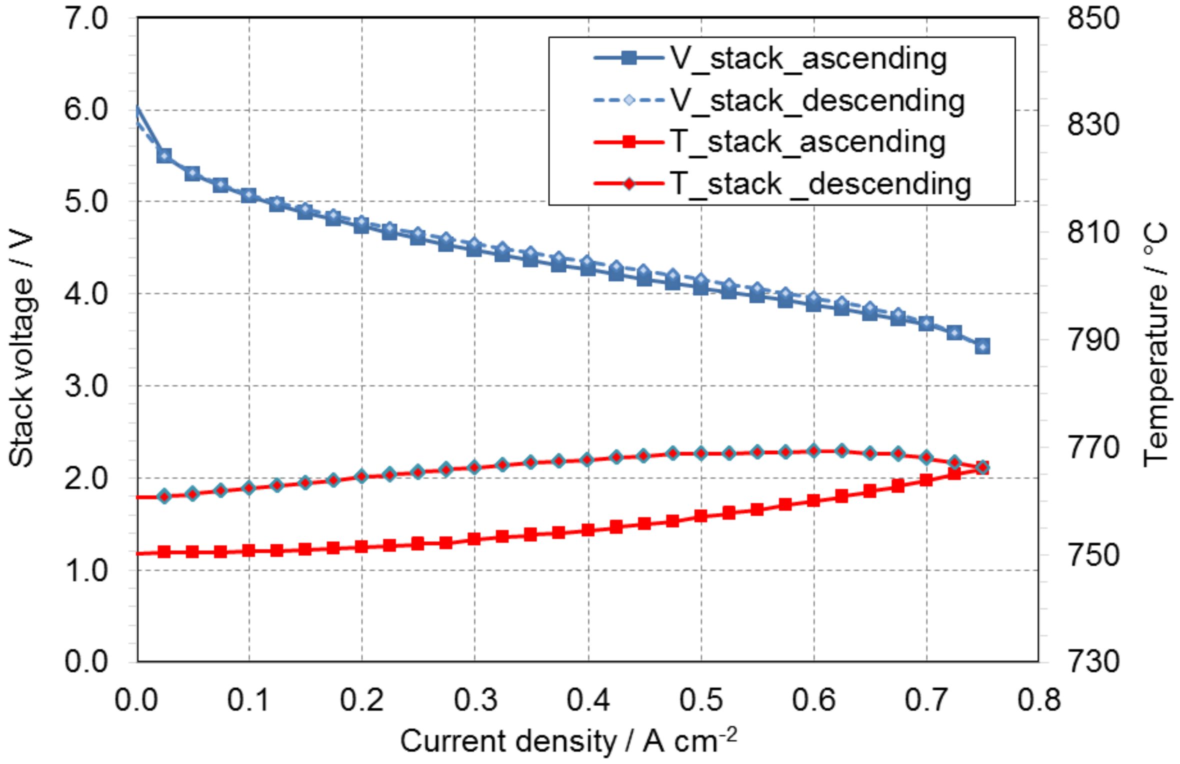


Figure 7: Example of a j-V curve measurement (ascending and descending) for an SOFC stack with 5 repeating units (RUs) including its (top plate) temperature

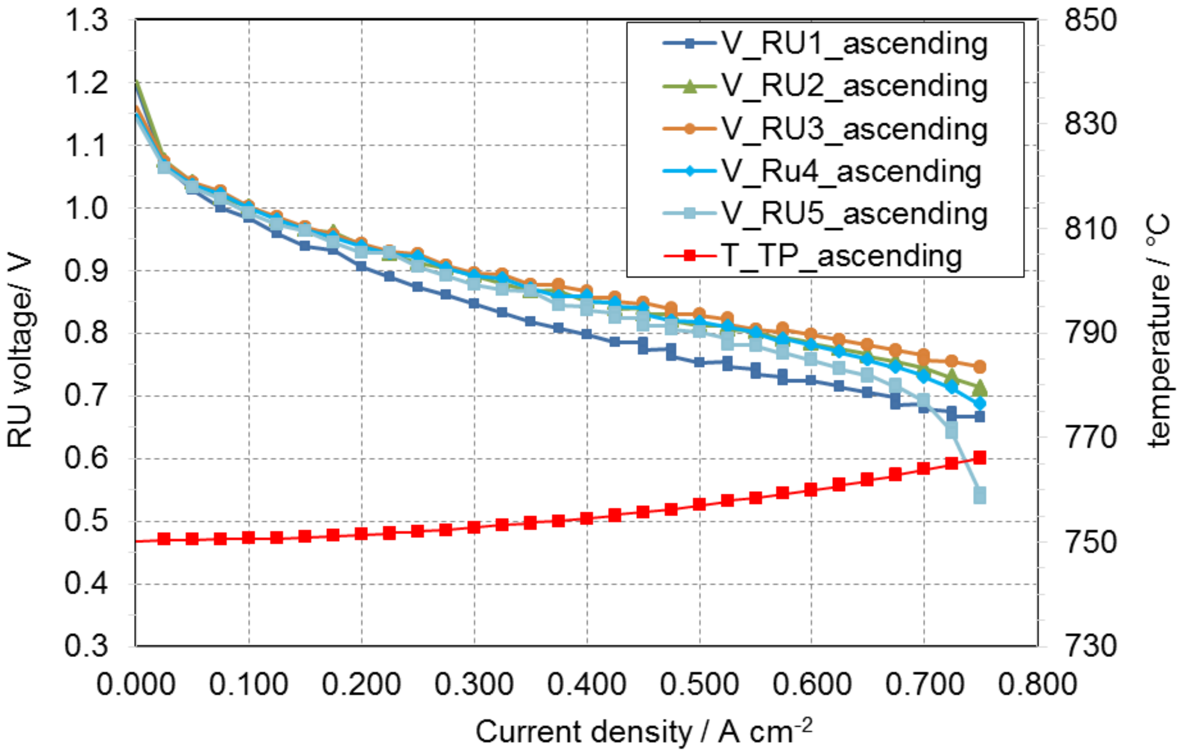


Figure 8: Example of the j-V curves (ascending parts) of the 5 repeating units (RUs numbered RU1 to RU 5 from bottom to top) of an SOFC stack (see Figure 7) including its (top plate) temperature

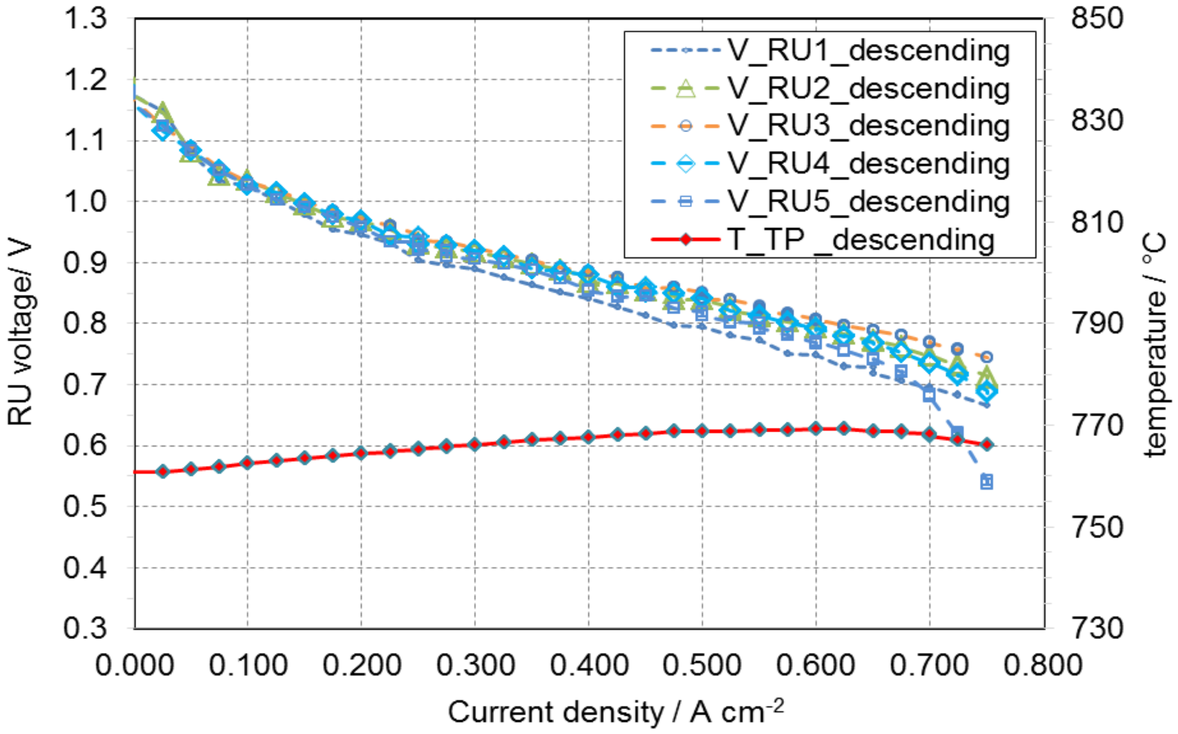


Figure 9: Example of the descending parts of the j-V curves for the 5 RUs (see Figure 8) including stack (top plate) temperature

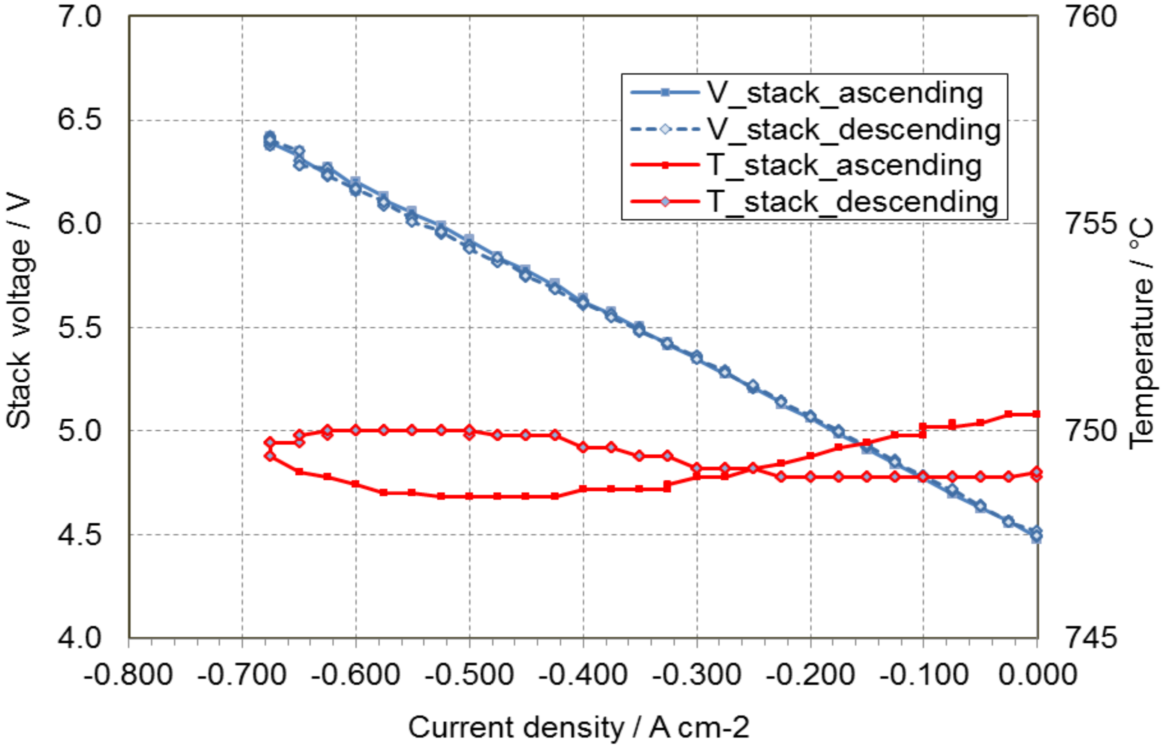


Figure 10: Example of a j-V curve measurement (ascending and descending) for an SOEC stack including its (top plate) temperature

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.

Test Module 04

Electrochemical Impedance Spectroscopy

(Version 3.9, 08 May 2017)

Objective and Scope

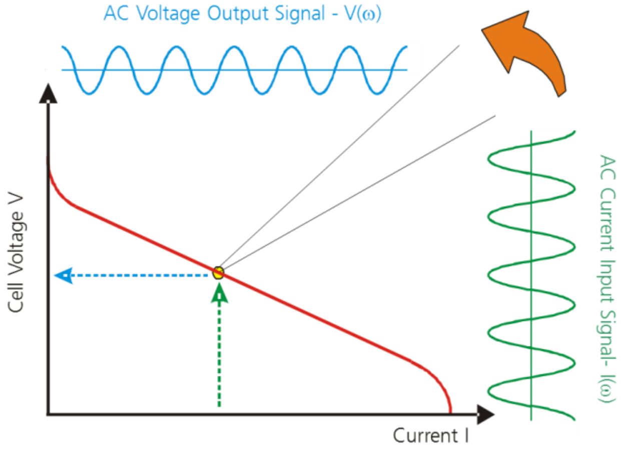
This test module deals with measuring of the electrochemical impedance spectra (EIS) of solid oxide cells (SOC) or stacks operated either as fuel cells (SOFC) or as electrolysers (SOEC). By varying the frequency of the sinusoidal excitation signal and measuring the corresponding response signal the different electrochemical processes taking place in the electrochemical system can be analysed. It is a general characterization method that can be used in SOC R&D and for quality assurance.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Direct current (*I*)	Frequency (υ)
Flow rates of inlet gases (f_{in})	
Temperature of the oven (T_{oven})	
Amplitude of alternating current ($\bar{I}$) or alternating voltage ($\bar{V}$)	
Composition of inlet gases ($x_{i,\ in}$)	
Pressure of outlet gases (p_{out})	

Test Procedure

- Connect the EIS system (waveform generator and frequency response analyser) according to the operating mode (SOFC, SOEC) to the test object.
- Impose the DC current under which the test object shall be examined.
- Impose an AC sinusoidal excitation signal (current or voltage) with small amplitude and measure the corresponding response signal (voltage or current).
- Choose an adequate number of measuring periods and number of measuring points per decade (compromise between measuring time and precision).
- Vary the frequency stepwise from high values (kHz) to identify the ohmic resistance to low values (mHz) to identify the overall resistance.



Input / Output signals of EIS

Critical Parameters and Parameter Controls

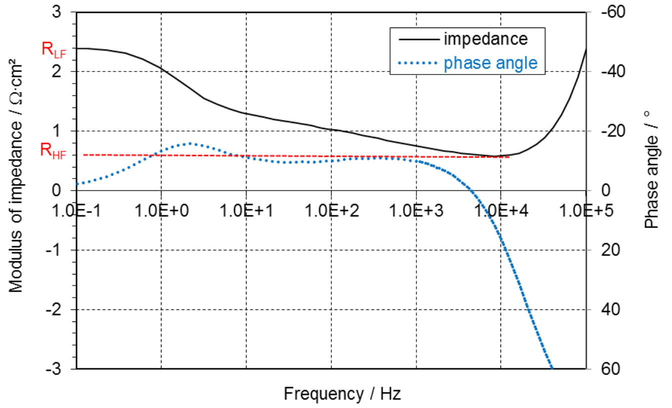
- Stability of all static TIPs is necessary to minimize voltage changes / fluctuations during the measurement.
- In SOEC mode, special attention is to be paid to a stable supply of steam to limit SOC voltage fluctuations to within a specified value e.g., +/-10 mV per cell or repeat unit.

Main Test Output Parameters (TOPs) and Derived Quantities

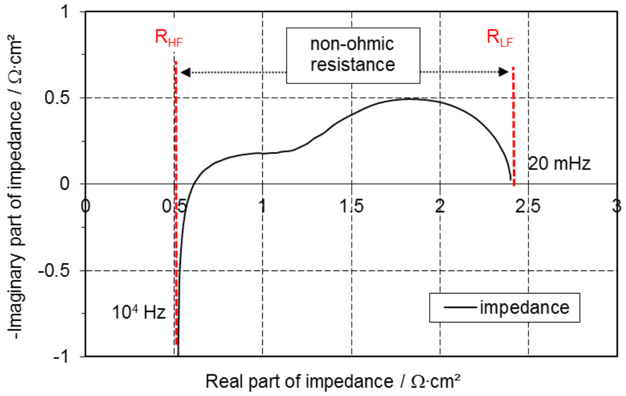
TOP	Derived Quantities
Alternating voltage (*V*) or alternating current (*I*) signal	Impedance (*Z*) Modulus of impedance ($\lvert Z\rvert$)
Phase angle (φ)	Real part of impedance (*Z′*)
Temperature of cell/stack (*T*)	Imaginary part of impedance (*Z″*)
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	Low frequency R_{LF} (or total) resistance
Voltage of cell / RU / stack (V)	High frequency R_{HF} (or ohmic) resistance

Data Post Processing and Representation

Representation examples of electrochemical impedance spectra:



Bode plot with modulus of impedance and phase angle vs. frequency



Cole-Cole plot representing imaginary part vs real part of impedance

SOCTESQA:

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Project website: www.soctesqa.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU Grant Agreement N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
Start date*: 01.05.2014 –* ***Duration****: 36 months*
Project Coordinator*: M. Lang – DLR*

Test Module 04: Electrochemical Impedance Spectroscopy

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Version: 3.9
Last revision: 08.05.2017

Content

Abbreviations

AC	Alternating current
ASR	Area specific resistance
DC	Direct current
ECM	Equivalent circuit model
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 04 – Electrochemical Impedance Spectroscopy

1 Objective and Scope

Electrochemical impedance spectroscopy (EIS) is an important tool to analyze processes taking place in electrochemical systems like fuel/electrolysis cells and stacks. It shows the different frequency dependent impedances of a test object which can either be a cell, a repeat unit of a stack or a complete stack. Moreover, it is used to understand the behaviour of the resistances of the cell/stack test object, e.g, the ohmic resistance, the polarization resistances of the electrodes and the gas conversion resistances.

However, there are only few documents on test procedures for this measurement method [1], [2]. Therefore, this test module contains important information on electrochemical impedance measurement and the resulting impedance spectra of SOC cells and stacks for both electrolysis and fuel cell modes. All important test parameters and conditions as well as a description on how to perform an impedance measurement are included in this test module. Additionally it provides information about the evaluation and analysis of the results.

Possible test objects which may be examined by electrochemical impedance spectroscopy are detailed shown in figures 4 and 5 of chapter 6.2 “Interface between test object and test environment” of TM 00 “General SOC Testing Guidelines” [3]. TM 00 also includes a comprehensive “list of quantities” including all symbols used in this test module.

2 Basic Principles

The fundamental approach of electrochemical impedance spectroscopy (EIS) is to apply a sinusoidal excitation signal with small amplitude to the system under investigation and measure the corresponding response signal (Figure 1). If the system satisfies contemporaneously the conditions of causality, linearity and time-invariance, the response to a sinusoidal current is a sinusoidal voltage and vice versa, both sharing the same frequency:

Input Signal: $$I(\omega, t) = \bar{I} \sin(\omega t) \qquad (1)$$

with $$\omega = 2\pi\upsilon \qquad (2)$$

An electrochemical system, e.g. a fuel or electrolysis cell/stack assembly, usually consists of elements with capacitive, inductive and/or resistive behaviour, which create phase angles/shifts between the alternating excitation signal and the corresponding response signal. Thus, the output signal can be formulated as follows:

Output Signal: $$V(\omega, t) = \bar{V} \sin(\omega t + \varphi) \qquad (3)$$

$\bar{I}$ = AC current amplitude
$\bar{V}$ = AC voltage amplitude
υ = frequency
ω = angular frequency
φ = phase angle/shift

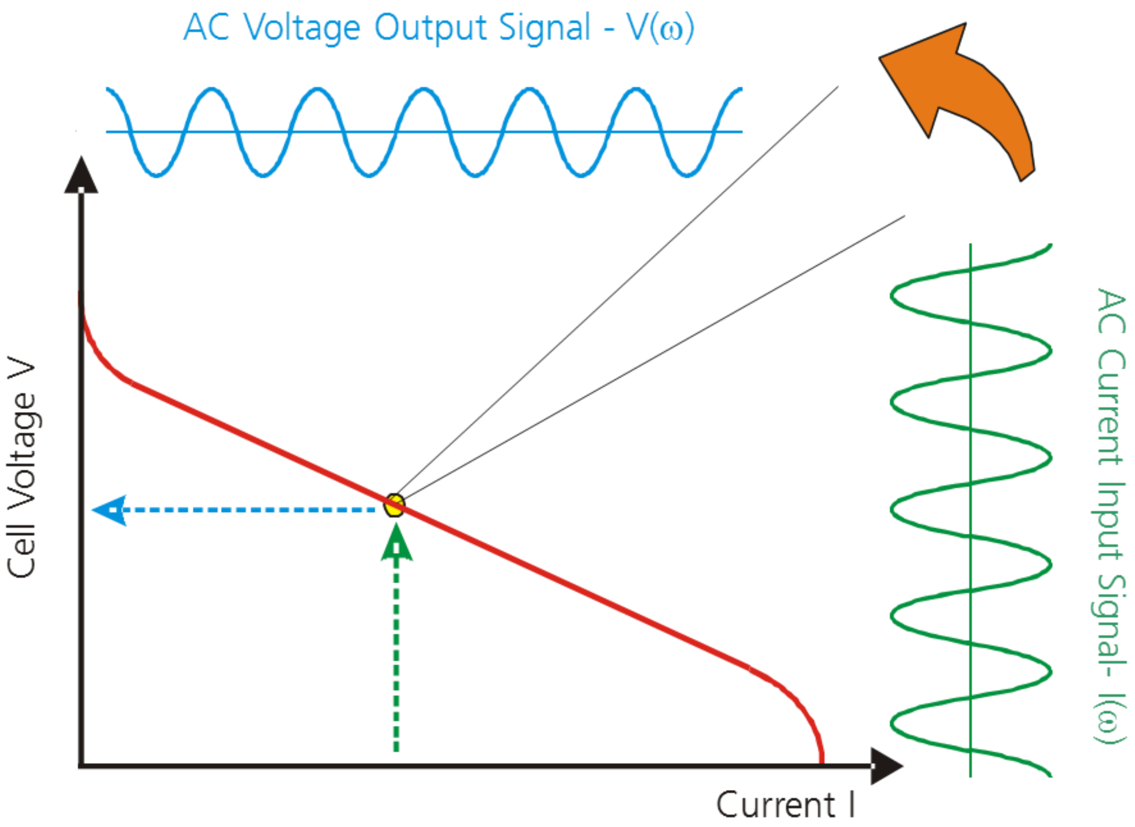

Figure 1: Input / Output signal of electrochemical impedance spectroscopy (EIS)

The impedance *Z(ω)* of an electrochemical component in the time domain is defined as ratio of voltage and current signal:

$$Z(\omega) = \frac{V(\omega,t)}{I(\omega,t)} = \frac{\bar{V}\sin(\omega t+\varphi)}{\bar{I}\sin(\omega t)} = |Z|\cdot\frac{\sin(\omega t+\varphi)}{\sin(\omega t)} \qquad (4)$$

The Fourier analysis converts a signal from its original domain (often time or space) to a representation in the frequency domain and vice versa:

$$Z(\omega) = \frac{FFT\{V(\omega,t)\}}{FFT\{I(\omega,t)\}} = |Z|\,exp(i\varphi) = |Z|\cos(\varphi) + |Z|\,i\sin(\varphi) = Z' + i\cdot Z'' \qquad (5)$$

The magnitude or the modulus of the impedance can be expressed:

$$|Z(\omega)| = \sqrt{Z'(\omega)^2 + Z''(\omega)^2} \qquad (6)$$

$$tan\varphi(\omega) = \frac{Z''(\omega)}{Z'(\omega)} \qquad (7)$$

Z' = real part of impedance

Z'' = imaginary part of impedance

$i^2 = -1$, imaginary unit property

The magnitude and the phase angle of the impedances of the cell/stack components change with the frequency. Therefore by varying the frequency of the excitation signal, the impedances of the studied electrochemical system can be determined as a function of frequency.

3 Test Equipment and Set-up

The impedance spectra can either be measured with alternating current (galvanostatic) or alternating voltage (potentiostatic) excitation input signal. If tests have to be performed in potentiostatic mode, an AC voltage waveform generator is needed. Otherwise, for galvanostatic test conditions, an AC current waveform generator is used. Furthermore, an AC voltage / current waveform analyzer (or frequency response analyzer, FRA) is required for recording the response of the cell or repeat unit. For measurements under DC operating current, an electrical load (fuel cell) or an additional power supply (electrolysis) is necessary. Figure 2 to Figure 5 show the generic set-ups for the 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 maybe 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 the whole stack whereas the voltage probes can either be connected to the complete stack or are just taken from the repeat unit of interest. The latter case has the advantage to examine the electrochemical behavior of individual repeat units of the stack. It is advised to minimize all kinds of high frequency artefacts, e.g. use twisted pair both for current and voltage probes, make sure that the voltage probes are perpendicular to the current probes etc.

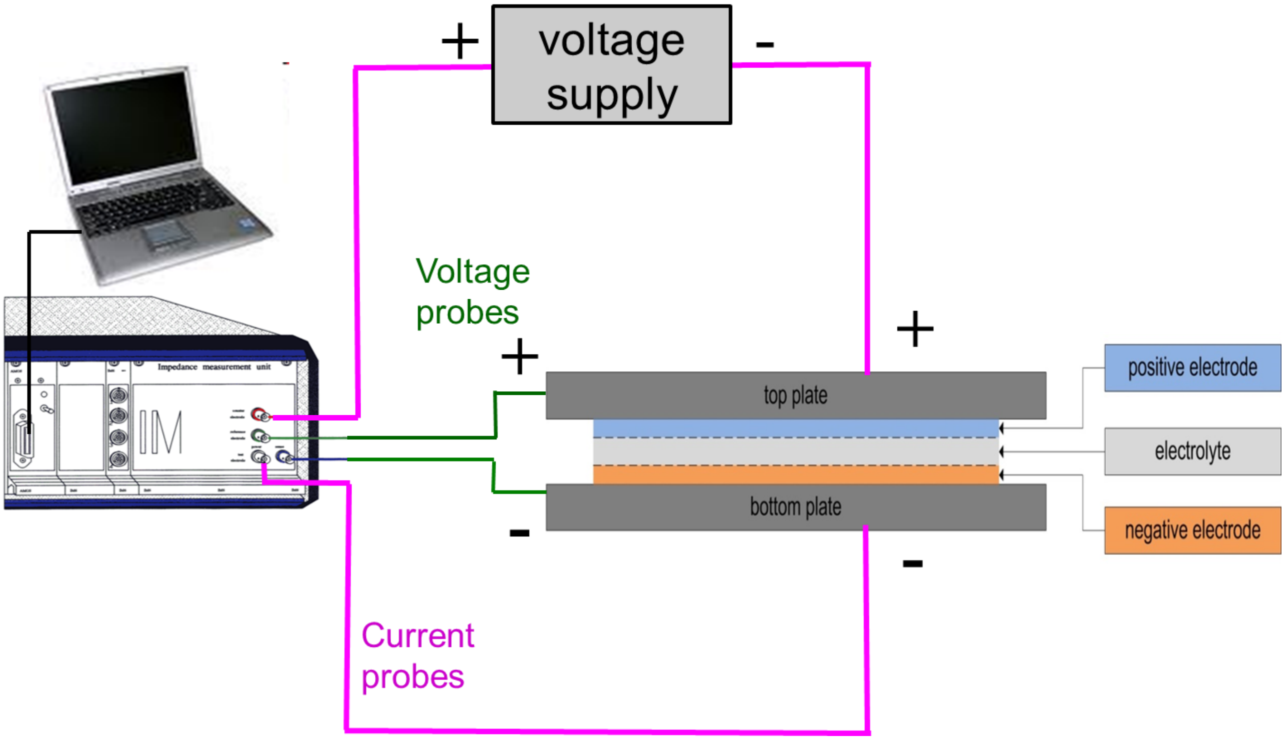

Figure 2: Set-up for electrochemical impedance spectroscopy of an SOFC cell

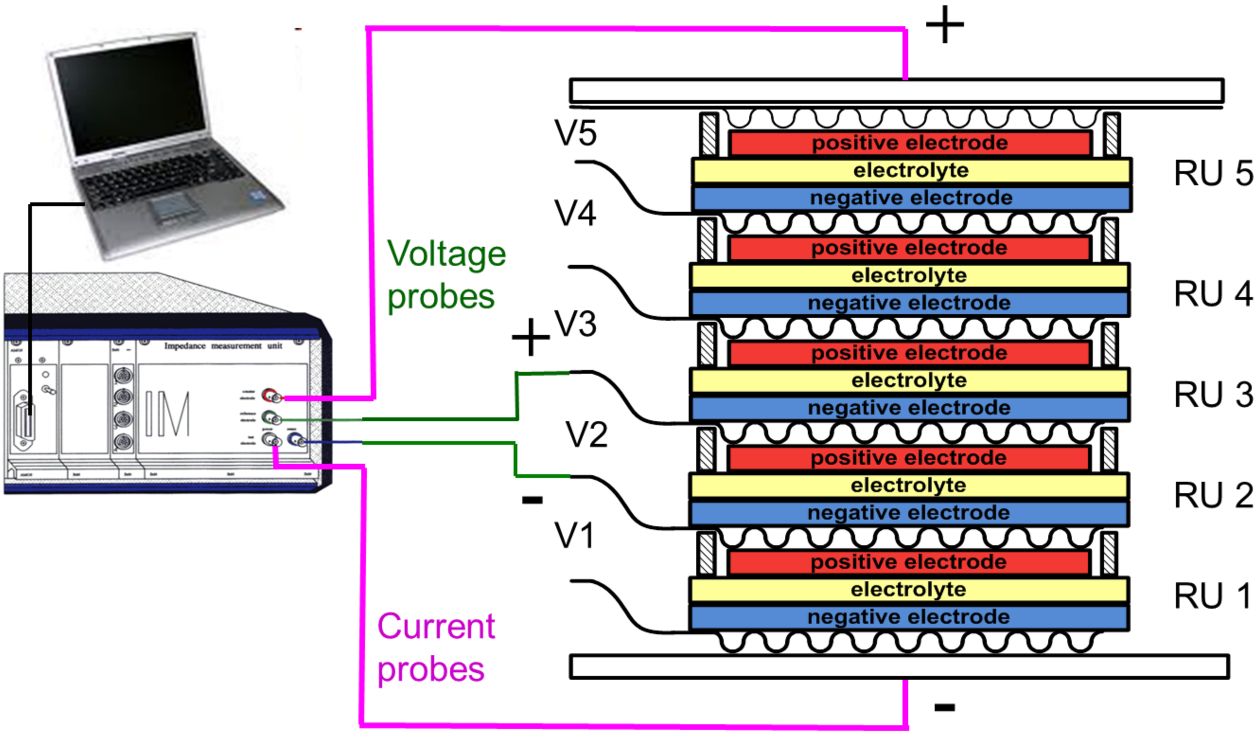

Figure 3: Set-up for electrochemical impedance spectroscopy of an SOFC stack

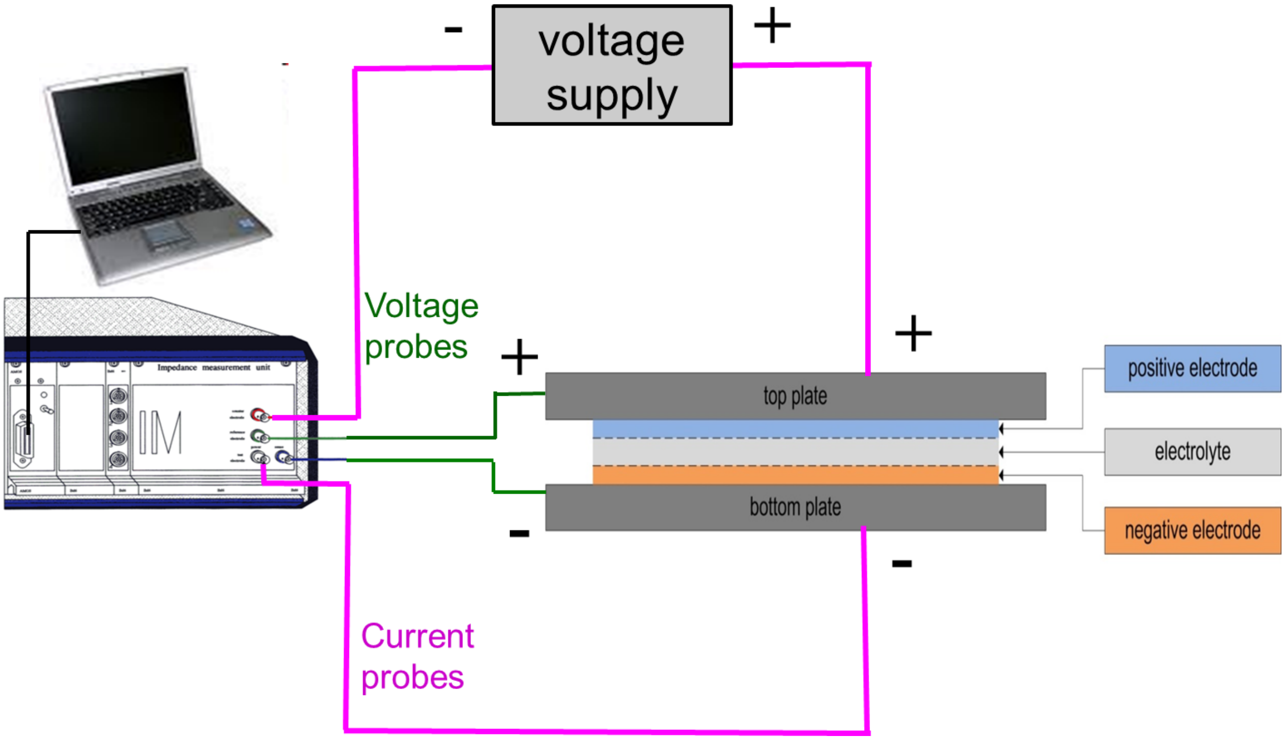

Figure 4: Set-up for electrochemical impedance spectroscopy of an SOEC cell

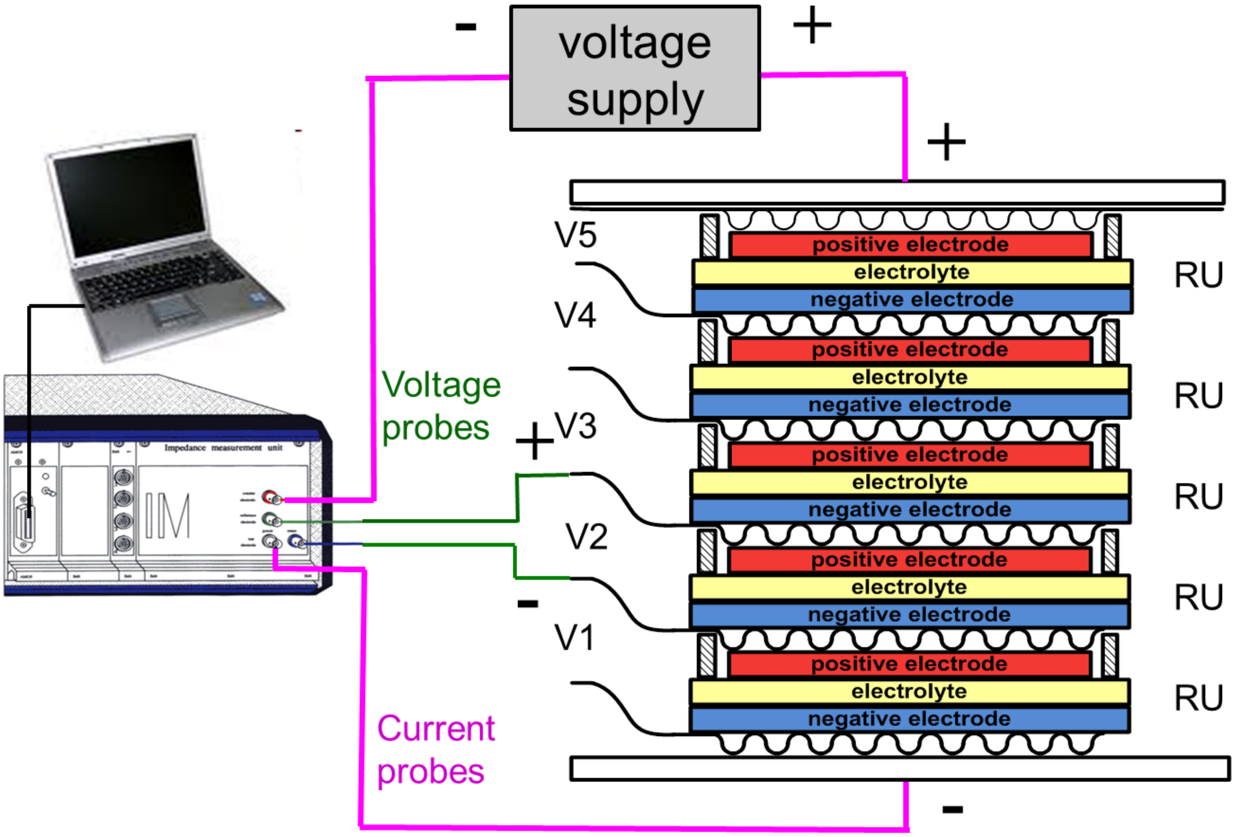

Figure 5: Set-up for electrochemical impedance spectroscopy of an SOEC stack

4 Test Input Parameters (TIPs)

There are two types of test inputs: variable and static. The first type may vary during the duration of the TM while the second type does not vary during the overall duration of the TM. The impedance spectra are usually measured in galvanostatic mode at a given constant DC current operating set point and with an alternating current input excitation signal. Another option is perform the measurement at a given direct voltage with an alternating voltage input excitation signal to the test object (potentiostatic mode). Table 1 lists all relevant static input parameters for performing electrochemical impedance spectra in galvanostatic mode. The parameters for potentiostatic mode are added in brackets. Table 2 shows the corresponding dynamic parameters which are varied during the measurement in galvanostatic mode (parameters for potentiostatic mode are added in brackets).

Table 1: Static test input parameters for electrochemical impedance spectroscopy

Description of quantity	Symbol	Unit often used	SI Unit
Active electrode area	A	cm²	m²
Compression force applied onto the cell/stack	F_{compr}	N	N
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}
Electrical direct current through the cell/stack	I	A	A
Electrical current density through the cell/stack	j	A cm^{-2}	A m^{-2}
Amplitude of alternating current in galvanostatic mode	$\bar{I}$	A	A
(Direct voltage of the cell/stack in potentiostatic mode)	(V)	(V)	(V)
(Amplitude of alternating voltage in potentiostatic mode)	$(\bar{V})$	(V)	(V)
Number of measuring periods for each measuring point for EIS spectra	k	-	-
Number of repeating units in the stack	N	-	-
Number of measuring points per decade for EIS spectra	n	-	-
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
Minimum frequency for EIS spectra	υ_{min}	Hz	Hz
Maximum frequency for EIS spectra	υ_{max}	Hz	Hz
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	$x_{i,pos,in}$	-	-

Table 2: Variable test input parameters for EIS

Description of quantity	**Symbol**	**Unit often used**	**SI Unit**
Frequency	υ	Hz	Hz

5 Test Output Parameters (TOPs)

Table 3 lists the test output parameters that are directly achieved by electrochemical impedance spectroscopy in galvanostatic mode. The parameters for potentiostatic mode are added in brackets.

Table 3: Test Output Parameters from EIS

Description of quantity	**Symbol**	**Unit often used**	**SI Unit**
Phase angle	φ	°	rad
Direct voltage of the cell in galvanostatic mode	V_{cell}	V	V
Direct voltage of repeating unit (RU) i in the stack in galvanostatic mode	$V_{RU,i}$	V	V
Direct voltage of the stack in galvanostatic mode	V_{stack}	V	V
(Direct current of the cell in potentiostatic mode)	(I_{cell})	(A)	(A)
(Direct current of repeating unit (RU) i in the stack in potentiostatic mode)	$(I_{RU,i})$	(A)	(A)
(Direct current of the stack in potentiostatic mode)	(I_{stack})	(A)	(A)
(Area specific) Impedance	Z	Ω, (Ω cm^2)	Ω
(Area specific) Modulus of impedance	$\lvert Z\rvert$	Ω, (Ω cm^2)	Ω
(Area specific) Real part of impedance	Z'	Ω, (Ω cm^2)	Ω
(Area specific) Imaginary part of impedance	Z''	Ω, (Ω cm^2)	Ω

6 Derived Quantities

The quantities listed in Table 4 are important and commonly used values calculated from input and output parameters. Additional derived quantities are described in the test module TM 00: “General SOC Testing Guidelines” [3].

Table 4: Derived quantities

Description of quantity	**Symbol**	**Unit often used**	**SI Unit**
(Area specific) low frequency resistance	R_{LF}	Ω, (Ω cm^2)	Ω
(Area specific) total oroverall resistance	R_{tot}	Ω, (Ω cm^2)	Ω
(Area specific) high frequency resistance	R_{HF}	Ω, (Ω cm^2)	Ω
(Area specific) ohmic resistance	R_{ohm}	Ω, (Ω cm^2)	Ω

7 Test Procedure

7.1 Critical parameters and parameter controls

The following test conditions shall be determined in advance by conducting preliminary tests when this test method is used:

1) DC current
EIS can be measured at open-circuit voltage and/or under electric load. A spectrum measured at OCV always means a change of the operation mode between SOFC and SOEC as the AC sinusoidal excitation signal has both positive and negative amplitude. Therefore it is recommended to perform the measurement at low DC current in the range of the AC amplitude to ensure performing the test in the desired operation mode. Measurements with higher DC current, e.g. at operation current density, are used to understand the different processes occurring under load. For electrolysis measurements the external current must be negative, therefore a DC current source is needed.

2) Amplitude of excitation signal
It is possible to excite either an alternating current or voltage. The amplitude of an AC input signal for the measurement should be high enough to generate a corresponding response signal with a high signal to noise ratio, but not too high in order to avoid measuring the spectrum in a nonlinear current - voltage response region. For using an AC voltage input signal the average amplitude per cell/RU is obtained by dividing the total voltage amplitude with the number of cells/RUs in series. It should be noted that in this case the AC voltage amplitudes applied on each RU could be different. The recommended perturbation amplitude for an AC current excitation signal is 3 - 30 mA/cm^2 and for an AC voltage excitation signal is 10 - 30 mV per cell depending on the system noise.

3) Measuring range of frequencies
The highest frequency should be high enough to identify the ohmic resistance (about 100 kHz) and the lowest frequency should be low enough to include the potential effects of gas diffusion and gas conversion in the cell/stack (about 30 mHz).

4) Sweep mode
It is recommended to start the measurements with high frequencies to get fast results and see directly if the measurement gives reasonable results. Afterwards sweep to lower frequencies.

5) Number of measuring points
Four to twenty points per order of frequencies (to be distributed evenly as logarithms, if possible) are required; they shall be numerous enough to identify clearly the geometry of impedance plots. If possible avoid the fundamental and harmonics of the electrical grid frequency.

6) Number of measuring periods
The measurement of the input signal is the average over more than one sine period. The larger the number of periods imposed, the longer the measurement will take. It is recommended to find a compromise between acceptable measuring time and precision.

7.2 *Preconditioning of the stack*

Before conducting the impedance measurement, the test conditions (e.g. operating temperature, fuel gases, oxidant gases, DC current) should be established as stable input parameters provide lower voltage fluctuations. It is important to precondition the stack until the temperature is stabilized. Especially when measuring spectra at high current densities a sufficient holding time is necessary after the current is increased to the set point in order to achieve thermal equilibrium.

In SOEC mode, special attention should be paid to the noise of voltage signals caused by insufficient stability in steam supply. The operation parameter of the steam generator should be optimised to reduce the noise to an acceptable level before starting the impedance measurement.

7.3 *Measurement of the electrochemical impedance spectrum*

The test shall be conducted using the following procedure:

1) Connect your cell/stack test object to the EIS system according to the required test mode (SOFC or SOEC) (see Chapter 3).
2) EIS can be measured at open-circuit voltage and/or under DC electric load. In the latter case increase the current stepwise to the set point. Precondition the cell/stack in order to achieve thermal equilibrium.
3) Verify that a steady state of your cell/stack in the test station has been reached. Moreover, voltage fluctuations should be kept as low as possible by ensuring stable input parameters, e.g. gas flow rates and electrical current load.
4) Superimpose AC sinusoidal waves of current or voltage at a characteristic (middle) frequency (e.g. 100 Hz) and make sure that a corresponding sinusoidal output signal is generated.
5) Start the measurement. Sweep the AC sinusoidal waves within the prescribed frequency range and measure the impedance at each selected frequency.
6) Save the measured data on a reliable data storage medium.

As an option, the validity of the impedance spectrum may be verified by using appropriate validation relations such as the “Logarithmic Kramers-Kronig (KK)” or “Z-hit” transform. These methods allow detecting violations of the EIS fundamentals of the examined system and the reconstruction of non-affected spectra, if the violations are not too strong [4].

8 Data Post Processing and Representation

In an EIS spectrum the directly achieved output parameter is the sinusoidal voltage (galvanostatic) or the sinusoidal current (potentiostatic). Moreover, the phase shift between the excitation signal and the corresponding response signal is measured directly as an output parameter. It is possible to obtain an electrochemical impedance spectrum by calculation of the corresponding impedances (see equation 4). One way is to calculate the total impedance as a function of frequency. Another possibility is to divide the total impedance (Z) into the imaginary part (Z'') and the real part (Z'). State-of-the-art impedance analysers contain powerful software packages, which directly supply these derived quantities as output parameters. Therefore, these impedance values can be treated as test output parameters (see Table 3).

There is more than one way to illustrate the results of impedance spectra in order to interpret the processes in the cell/stack. A Bode plot shows for example the phase angle and the modulus of impedance (see Figure 6) as a function of frequency. Another option is to plot the real and imaginary part (see Figure 7) as a function of frequency. Assuming the impedance reaches a constant level at low frequencies, the low-frequency resistance R_{LF} can be easily read from this graph. This represents the overall impedance of the test object, the ASR (area specific resistance). The lowest point of the impedance shows the high-frequency resistance R_{HF} which can be seen as the ohmic resistance of the cell/stack. The changes of the phase angle and the slope of the impedance are related to the different loss processes appearing at different frequency ranges.

In a Cole-Cole diagram - often also called Nyquist diagram - the conjugated imaginary and real parts of the impedance are plotted (see Figure 8). In this case, the low-frequency resistance R_{LF} is given by the (second) intersection of the curve with the x axis. The intersection of the tangent drawn to the curve with the x axis shows the value of R_{HF}. The difference of the high frequency resistance and the low frequency resistance gives the non-ohmic resistance of the cell/stack. This value usually contains the polarization impedances and the gas conversion impedances of the positive and negative electrodes. In the Cole-Cole diagram the arcs are caused by frequency dependent processes, e.g. the electrochemical process at the electrodes and gas concentration processes of the electrodes.

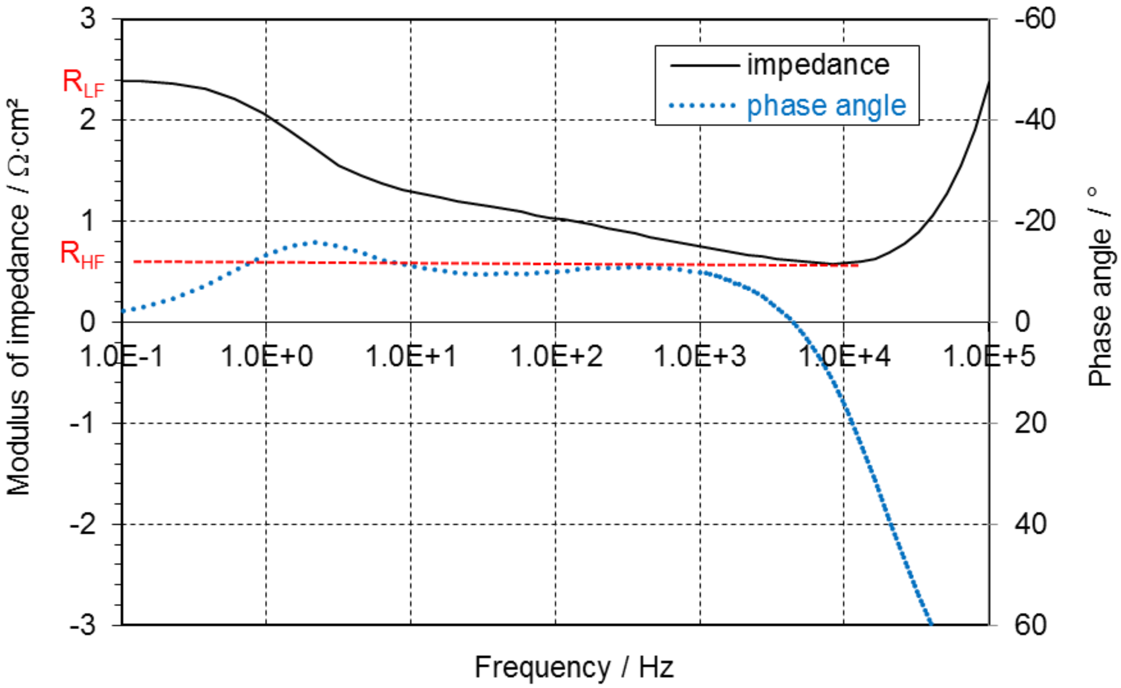

Figure 6: Bode plot representing the modulus of impedance and phase angle vs. frequency

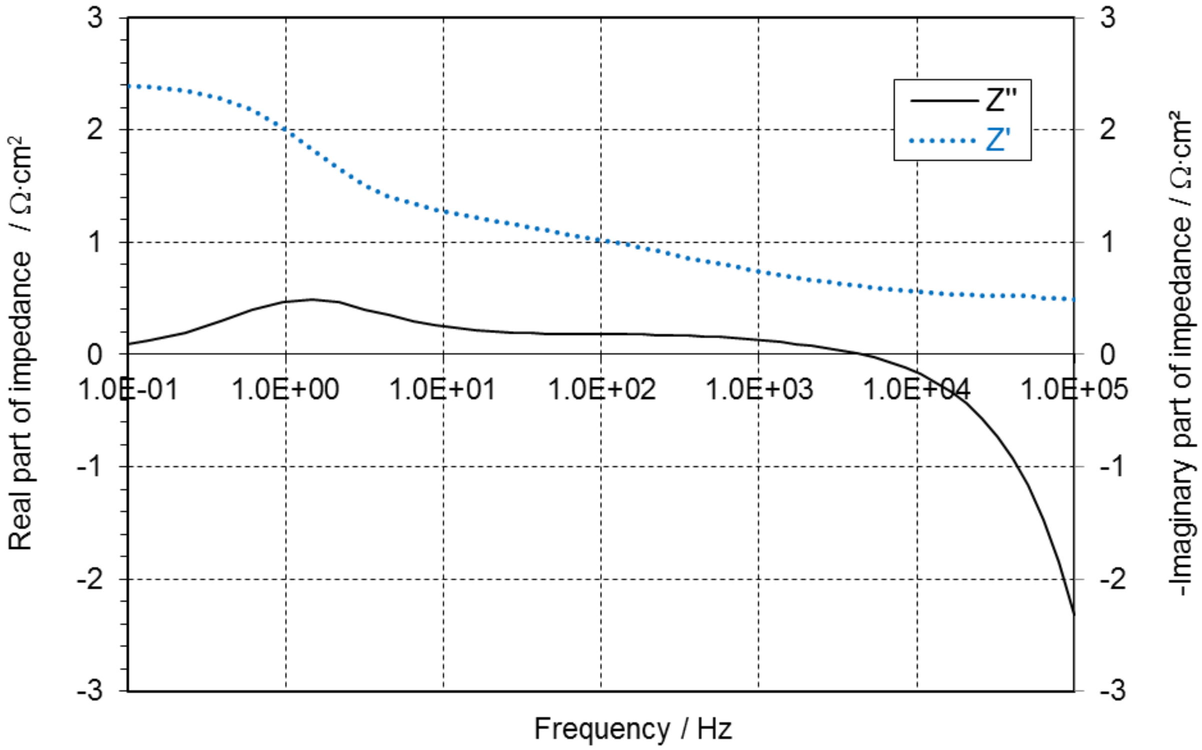

Figure 7: *Bode plot representing real and imaginary part of impedance vs. frequency*

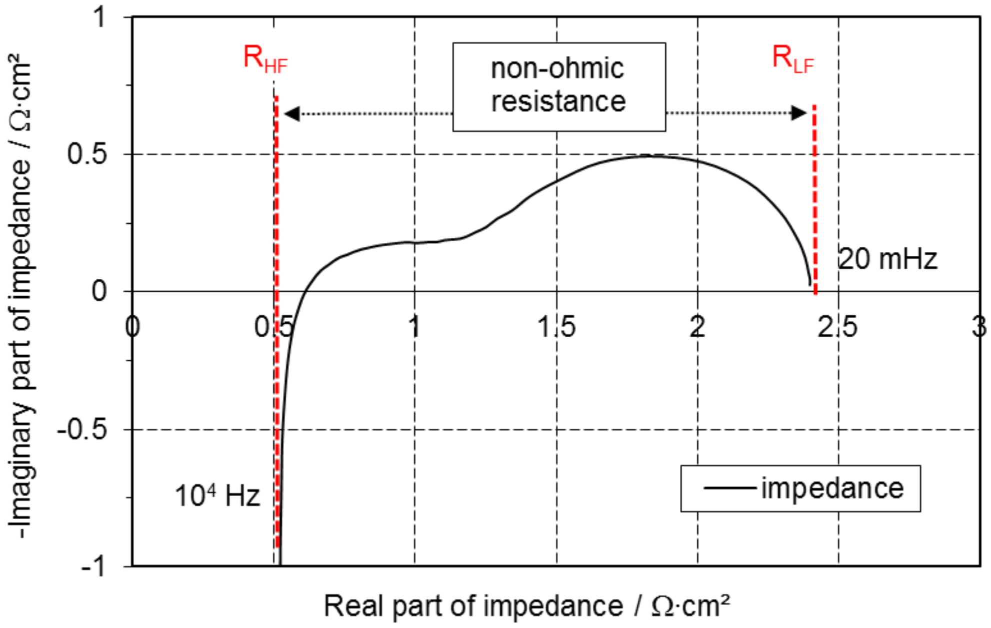

Figure 8: Cole-Cole plot representing imaginary part vs real part of impedance

The physicochemical and electrochemical processes taking place in a fuel/electrolysis cell or stack repeat unit can be modeled as an equivalent circuit consisting of passive elements such as resistors, capacitors and inductances. Factually, these processes are distributed in space and time hence special circuit elements describing these realistic phenomena are usually taken into consideration. For instance, the Warburg element is often employed to represent the gas diffusion impedance of the SOC electrodes while the generalized constant phase element (CPE) usually replaces the ideal capacitor. Each element of the equivalent circuit represents a physicochemical or electrochemical process taking place in the cell/stack layer and is excited at a particular characteristic time. There are many different equivalent circuit models (ECMs) which are described in literature, for example in [5], [6], yet there is discrepancy on how these should be chosen/built. Different equivalent circuit models have shown to have virtually identical frequency dependent impedance behaviour.

After choosing a suitable equivalent circuit model, initial values are assigned to the elements in the model. These values can be determined in different ways. The starting values for the model can be read out from the Cole-Cole or Bode plot. The initial values are provided as input to the simulation model. Using complex nonlinear least squares (CNLS) algorithm, software can generate a curve (which is referred as a "fit”) for simulating the experimental result. At the end of the simulation, the values for the various resistors, capacitors and the inductor are identified.

Results from impedance spectra fitting should be analyzed with caution. Firstly, they represent complicated electrochemical phenomena taking place inside the layers of the cell/stack unit, which have often not been completely understood. Secondly, the spectra are usually very sensitive to measuring conditions especially temperature, current and gas composition. Thirdly, disturbances such as high frequency disturbance from measuring lines influence or handicap the measurement. Fourthly, fitting of the spectra to the model is not error free – the nature of the curve with the overlap between the different impedance arcs (i.e. the frequency overlap between different electrochemical processes) as well as human induced error can give different results even when a single curve is being fitted twice by the same person. Because of these reasons, it is sometimes more advisable to use the direct results from the EIS spectra to analyze the impedance changes in relation to the different cell/stack layers, operating conditions or as a function of time.

9 Differences to Existing Procedures

Up to now only a brief description for measuring electrochemical impedance spectra on SOFC cells/stacks is given in the IEC document “IEC 62282-7-2, Part 7-2” [1]. This document describes the procedure for measuring EIS spectra very briefly and the presentation of the results with Cole-Cole plots. Another document describes several test methods (voltammetry, potentiometry and electrochemical impedance spectroscopy) for polymer electrolyte membrane stacks [2]. This document includes the setup of the test, the procedure and the data representation specifically for low temperature fuel cell stacks.

In contrast to the above mentioned documents, the present test module TM04 describes much more detailed the electrochemical impedance spectroscopy for solid oxide cells and stacks. The basic principles of EIS, the test equipment and setup, the relevant test input and output parameters, the derived quantities, the test procedure for measuring EIS spectra are reported. Moreover, the data post processing and representation of the results with all important diagrams and interpretation of the results are explained. Finally, the present test module document addresses both the fuel cell (SOFC) and the electrolysis (SOEC) operation modes.

10 Bibliography

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Test Module 07

Reactant Utilisation

(Version 5.1, 09 May 2017)

Objective and Scope

The purpose of this test module is to characterize the influence of the reactant utilisation on the performance of a Solid Oxide Cell (SOC) cell/stack either in fuel cell (SOFC) mode or in electrolysis (SOEC) mode. The parameters governing the reactant utilisation are the current generated (SOFC) or consumed (SOEC) by the cell/stack, the gas composition and the volumetric flow rate of the fuel/oxidant gas stream.

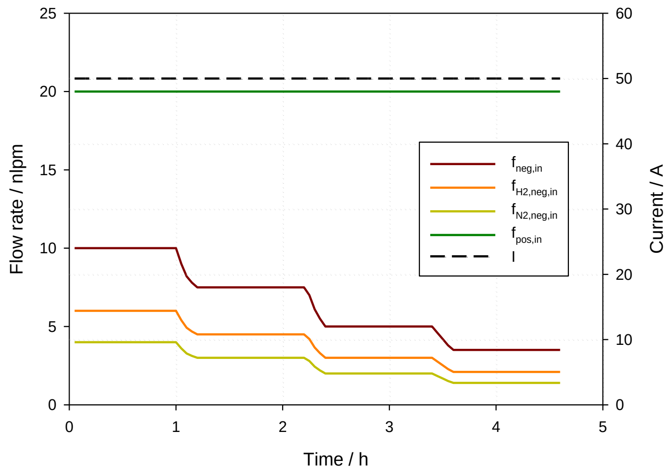
It must be noted that TM07 only contains the variation of the volumetric flow rate of the fuel/oxidant gas streams because the variation of the electrical current is already described in TM03: *Current Voltage Characteristics* and the effect of the gas composition variation is already described in TM08: *Reactant Gas Composition.*

Main Test Input Parameters (TIPs)

Static TIP	**Variable TIP**
Current (*I*)	Flow rates of inlet gases (f_{in})
Temperature of the oven (T_{oven})	
Composition of inlet gases ($x_{i,\ in}$)	

Test Procedure

- The operating conditions of the cell/stack, including the temperature, the electrical current and the reactant gas composition will be defined beforehand and will be kept constant throughout the whole TM07. Only the volumetric flow rates of the negative or positive electrodes will be varied, being this variation discrete and only happening in one electrode at a time.
- It is recommended to start the test with low fuel utilisations (i.e. high volumetric flows) and not to surpass the maximum reactant utilisation recommended by the manufacturer.



Qualitative representation of evolution of TIPs for instance in SOFC mode when carrying out TM07 varying $f_{neg,in}$.

Critical Parameters and Parameter Controls

- For all test conditions care should be taken not to incur reactant starvation.
- The test campaign will begin with the most benign operating conditions (low gas utilizations) and will shift towards harsher conditions.

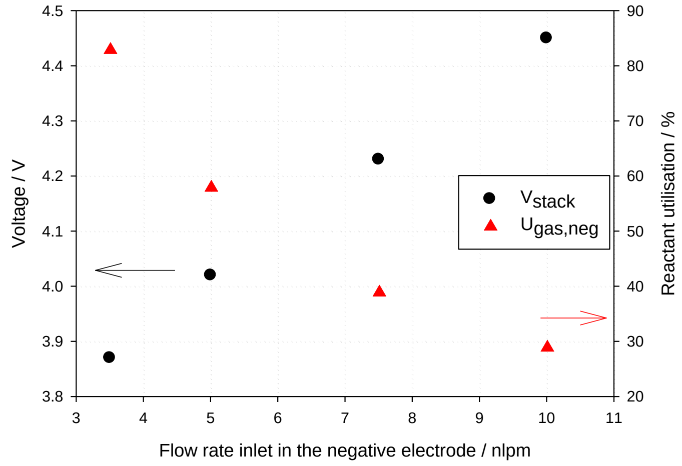
TIPs	**Direction of change**
$f_{neg,in}$ or $f_{pos,in}$	$HIGH\ \rightarrow LOW$

Main Test Output Parameters (TOPs) and Derived Quantities

TOP	**Derived Quantities**
Voltage of cell/RU/stack (*V*)	Current density (*j*)
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (*T*)	Electrical power density ($P_{d,el}$)
Flow rates of outlet gases (f_{out})	Reactant gas utilization (U_{gas})

Data Post Processing and Representation

The figure below is an example of how to correlate graphically the TIPs and TOPs of this test module. In this particular example, the average cell voltage has been the chosen TOP. Nevertheless, others could be plotted, such as the various temperatures in the stack.



Example graph of representation of results in SOFC mode when varying $f_{neg,in}$.

SOCTESQA:
Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Project website: www.soctesqa.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU GRANT AGREEMENT N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
***Start date**: 01.05.2014 – **Duration**: 36 months*
***Project Coordinator**: M. Lang – DLR*

Test Module 07: Reactant Utilisation

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Content

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 07 – Reactant Utilisation

1 Objective and Scope

The purpose of this test module (TM) is to characterize the influence of the reactant utilisation on the performance of a Solid Oxide Cell (SOC) cell/stack either in fuel cell (SOFC) mode or in electrolysis (SOEC) mode. The parameters governing the reactant utilisation are the current generated (SOFC) or consumed (SOEC) by the cell/stack, the gas composition and the volumetric flow rate of the fuel/oxidant gas stream.

It must be noted that TM07 only contains the variation of the volumetric flow rate of the fuel/oxidant gas streams because the variation of the electrical current is already described in TM03: *Current Voltage Characteristics* and the effect of the gas composition variation is already described in TM08: *Reactant Gas Composition.*

As an example, in SOFC mode the reagents are hydrogen (negative electrode) and oxygen (positive electrode) while the product is steam (negative electrode). In SOEC mode the reagent is steam (and possibly CO_2, negative electrode) and the products are hydrogen (and possibly CO, negative electrode) and oxygen (positive electrode). In SOEC mode the reactant utilisation at the negative electrode is usually called steam conversion rate.

This test procedure has no specific target application. However, this test module describes a general characterization method that can be used in research and development of the SOC and for quality assurance in cell and stack production. This module is applied in combination with other test modules in a test programme. The test programme will be application-specific and also describes the operating conditions of the SOC in more detail. All the quantities used in TM07 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.

Note: the parameters, values and range of values including uncertainties used throughout this document are recommended only.

2 Test Equipment and Set-up

This part is fully detailed in chapter 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)

There are two types of test input parameters (TIPs): variable TIPs and static TIPs (for definitions refer to Table 1 in TM00). The first type may vary during the duration of the TM while the second type does not vary during the overall duration of the TM. The SOC assembly units are usually operated in galvanostatic mode (at a constant current for a given operation point).

The measurement uncertainties, the sampling rates and the values are given in the master document TM00. Deviations from the test module should be reported in the test report.

3.1 SOC – Variable flow rate of the negative electrode gas stream at cell/stack inlet ($f_{neg,in}$)

The static TIPs for this test are listed in Table 1. Table 2 lists the variable TIPs for the case when the only variable parameter is the flow rate of the negative electrode gas stream at cell/stack inlet.

In order to evaluate the effect of the flow rate of the negative electrode gas stream at cell/stack inlet, the molar fraction of the reactant in the negative electrode must be kept constant, hence the ratio $f_{i,neg,in}$ / $f_{neg,in}$ must be kept constant (i.e. constant $x_{i,neg,in}$).

Table 1: Static test input parameters during the TM07 – SOC test mode varying $f_{neg,in}$

Description of quantity	Symbol	Unit often used	SI Unit
Electrical current through the cell/stack	I	A	A
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 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	$x_{i,pos,in}$	-	-
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

Table 2: Variable test input parameters during the TM07 – SOC test mode varying $f_{neg,in}$

Description of quantity	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 the negative electrode gas stream at cell/stack inlet	$f_{neg,in}$	nlpm, slpm l_n min^{-1}, l_s min^{-1}	$m^3 s^{-1}$

3.2 SOFC – Variable flow rate of the positive electrode gas stream at cell/stack inlet ($f_{pos,in}$)

This method is equivalent to that of section 3.1, but is applied to the positive electrode gas stream. The static TIPs for this test are listed in Table 3. Table 4 lists the variable TIPs for the case when the only variable parameter is the flow rate of the positive electrode gas stream at cell/stack inlet. Usually air is fed to the positive cathode, as a single, pre-mixed compound gas.

Table 3: Static test input parameters during the TM07 – SOFC test mode varying $f_{pos,in}$

Description of quantity	Symbol	Unit often used	SI Unit
Electrical current through the cell/stack	I	A	A
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 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}
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	$x_{i,pos,in}$	-	-
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

Table 4: Variable test input parameters during the TM07– SOFC test mode varying $f_{pos,in}$

Description of quantity	Symbol	Unit often used	SI Unit
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}
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}

4 Test Output Parameters (TOPs)

The main recorded output parameters of the tests described above are the voltage, the flows exiting the cell/stack and the temperatures. All of these are tabulated in Table 5.

Table 5: Test output parameters in TM07 for SOFC and SOEC modes as defined in TM00

Description of quantity	**Symbol**	**Unit often used**	**SI Unit**
Voltage of the stack	V_{stack}	V	V
Voltage of repeating unit (RU) *i* in the stack	$V_{RU,i}$	V	V
Voltage of the cell	V_{cell}	V	V
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	$x_{i,pos,out}$	-	-
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
Pressure of the negative electrode gas stream at cell/stack inlet	$p_{neg,in}$	mbar, kPa	N m^{-2} (Pa)
Pressure of the positive electrode gas stream at cell/stack inlet	$p_{pos,in}$	mbar, kPa	N m^{-2} (Pa)

5 Derived quantities

The reactant utilisation of the negative and positive electrodes gas streams can be calculated according to Eq. (1) and Eq. (2).

$$U_{gas,neg} = \frac{I \times N}{71.74 \times \sum_{i=1}^{n} z_i \times f_{i,neg,in}} \times 100\% \tag{1}$$

$$U_{gas,pos} = \frac{I \times N}{71.74 \times \sum_{i=1}^{n} z_i \times f_{i,pos,in}} \times 100\% \tag{2}$$

I is the electrical current, N is the number of repeat units, z_i is the number of electrons of reactant i participating in the electrochemical reaction and f_i is the flow rate of reactant i of the gas stream at cell/stack inlet expressed in nlpm.

Usually for SOFC the reactant components at the negative electrode are H_2, CO, CO_2, CH_4 and at the positive electrode O_2. In SOEC mode the reactant components at the negative electrode usually are H_2O and CO_2.There is no reactant utilisation at the positive electrode in SOEC mode.

The parameters listed in Table 6 are important and commonly used derived quantities calculated from input and output parameters.

Table 6: Derived quantities possibly calculated during the TM07

Description of quantity	Symbol	Unit often used	SI Unit
Electrical current density through the cell/stack	j	A cm^{-2}	A m^{-2}
Electrical power of the stack	P_{el}	W	J s^{-1} (W)
Gas utilization at the negative electrode	$U_{gas,neg}$	%	-
Gas utilization at the positive electrode	$U_{gas,pos}$	%	-
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)

6 Test Procedure

6.1 Critical Parameters and Parameter Controls

6.1.1 Reactant starvation

For all test conditions care should be taken not to incur reactant starvation.

6.1.2 Degradation induced by tests

The test campaign will begin with the most benign operating conditions (low gas utilizations) and will shift towards harsher conditions (Table 7).

Table 7: Critical TIPs and parameter control

Test input parameters	Direction of change
$f_{neg,in}$ or $f_{pos,in}$	$HIGH \rightarrow LOW$

6.2 Preconditioning of the Stack

The preconditioning of the fuel cell stack is specified in TM00 and should be realized under nominal conditions given by the stack manufacturer. It must be noted that temperature and flow gas stabilisations are of utmost importance for the tests described in this test module.

6.3 Test for Reactant Utilisation

6.3.1 Constraints and considerations

The ranges for the reactant utilisations – Eq. (3) and Eq. (4) – as well as the operating conditions have to be defined beforehand taking into consideration the recommendations provided by the manufacturer concerning the operation of the stack.

$$U_{gas,neg}^{MIN} \leq U_{gas,neg} \leq U_{gas,neg}^{MAX} \tag{3}$$

where $U_{gas,neg}^{MIN}$ and $U_{gas,neg}^{MAX}$ are the minimum and maximum negative electrode gas utilisations respectively suggested by the stack manufacturer.

$$U_{gas,pos}^{MIN} \leq U_{gas,pos} \leq U_{gas,pos}^{MAX} \tag{4}$$

where $U_{gas,pos}^{MIN}$ and $U_{gas,pos}^{MAX}$ are the minimum and maximum positive electrode gas utilisations respectively suggested by the stack manufacturer.

6.3.2 Methodologies

The operating conditions of the cell/stack, including the temperature, the electrical current and the reactant gas composition will be defined beforehand and will be kept constant throughout the whole TM07. Only the volumetric flow rates in the negative or positive electrodes will be varied, being this variation discrete and only happening in one electrode at a time. It is recommended to start the test with low fuel utilisations (i.e. high volumetric flows) and not to surpass the maximum reactant utilisation recommended by the manufacturer.

Example 1 – SOFC ($U_{gas,neg}$)

Table 8: Test matrix example illustrating the most relevant variable (highlighted) and static TIPs to be used in the test in order to address the impact of fuel utilisation (i.e. $U_{gas,neg}$) on a 5-cell stack when diluted hydrogen is used as fuel.

$U_{gas,neg}$ (%)	***I* (A)**	**$f_{neg,in}$ (nlpm)**	**$x_{H2,neg,in}$ (-)**	**$f_{pos,in}$ (nlpm)**	**$x_{O2,pos,in}$ (-)**	**$U_{gas,pos}$ (%)**
29	50	**10**	0.6	20	0.21	21
39	50	**7.5**	0.6	20	0.21	21
58	50	**5**	0.6	20	0.21	21
83	50	**3.5**	0.6	20	0.21	21

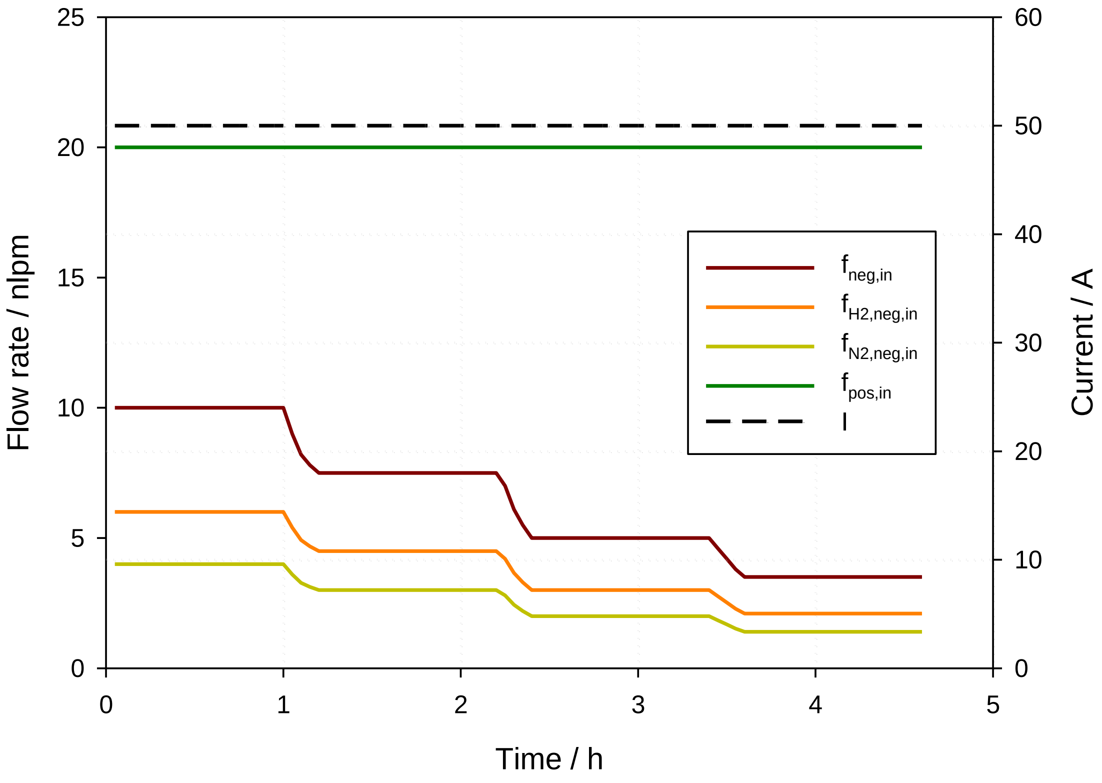


Figure 1: Qualitative representation of TIPs for instance in SOFC mode when carrying out TM07 varying $f_{neg,in}$.

Example 2 – SOFC ($U_{gas,pos}$)

Table 9: Test matrix example illustrating the most relevant variable (highlighted) and static TIPs to be used in the test in order to address the impact of oxygen utilisation (i.e. $U_{gas,pos}$) on a 5-cell stack when air is fed to the positive electrode.

$U_{gas,pos}$ (%)	***I* (A)**	**$f_{pos,in}$ (nlpm)**	**$x_{O2,pos,in}$ (-)**	**$x_{H2,neg,in}$ (-)**	**$f_{neg,in}$ (nlpm)**	**$U_{gas,neg}$ (%)**
21	50	**20**	0.21	0.6	7.5	39
28	50	**15**	0.21	0.6	7.5	39
42	50	**10**	0.21	0.6	7.5	39

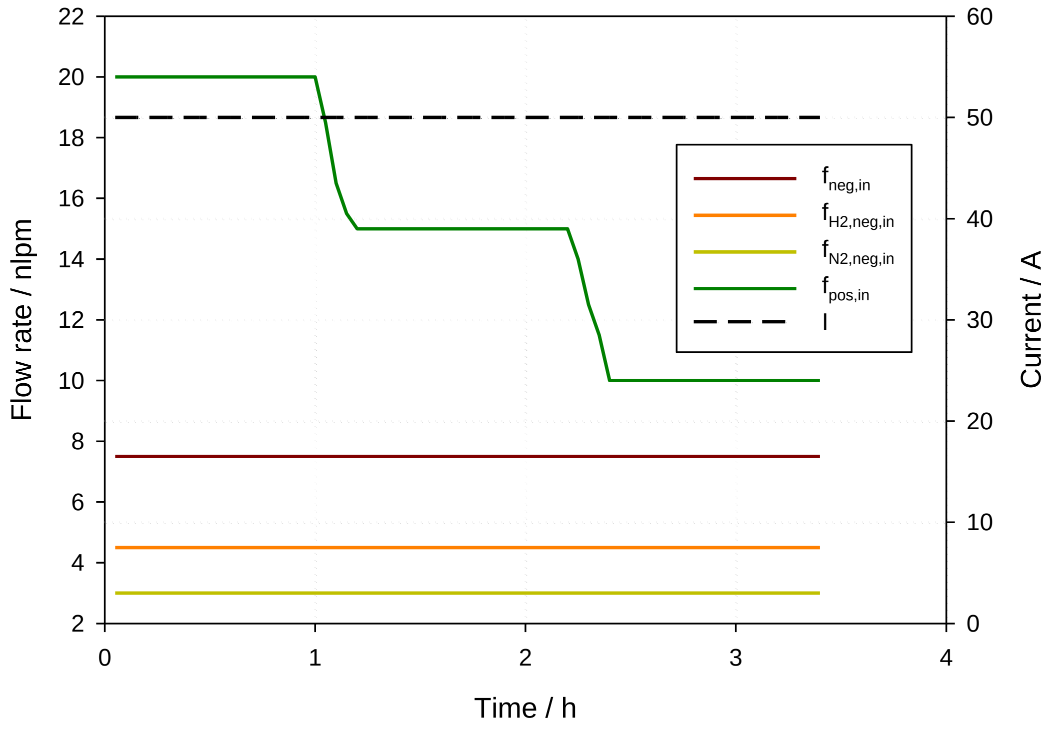


Figure 2: Qualitative representation of TIPs for instance in SOFC mode when carrying out TM07 varying $f_{pos,in}$

Example 3 – SOEC

Table 10: Test matrix example illustrating the most relevant variable (highlighted) and static TIPs to be used in the test in order to address the impact of steam conversion (i.e. $U_{gas,neg}$) on a 5-cell stack.

$U_{gas,neg}$ (%)	I (A)	$f_{neg,in}$ (nlpm)	$x_{H2O,neg,in}$ (-)	$f_{pos,in}$ (nlpm)
29	-50	**10**	0.6	20
39	-50	**7.5**	0.6	20
58	-50	**5**	0.6	20
83	-50	**3.5**	0.6	20

6.3.3 Stabilization times

For stabilization times, please refer to the cell or stack manufacturer/supplier. In case a stabilization period is not provided, the procedure described in TM00 (Chapter 6.5) can be used.

7 Data Post Processing and Representation

Equations (1) and (2) enable calculating the reactant utilisation in the negative and positive electrodes respectively.

In SOFC mode the reactant utilisation can be used to calculate the volumetric flow of steam being produced in the negative electrode. If steam can be condensed in the experimental set-up, then the difference between the measured volume of water over a predetermined period of time and its theoretical value can give an insight of the gas losses inside the stack.

Additionally, in SOEC mode the reactant utilisation can be used to calculate the volumetric flow of hydrogen being produced in the negative electrode and of oxygen in the positive electrode.

Figure 3 is an example of how to correlate graphically the TIPs and TOPs of this test module. In this particular example the average cell voltage has been the chosen TOP; nevertheless, others could be plotted, such as the various temperatures in the stack or the pressure differences in the positive and negative streams.

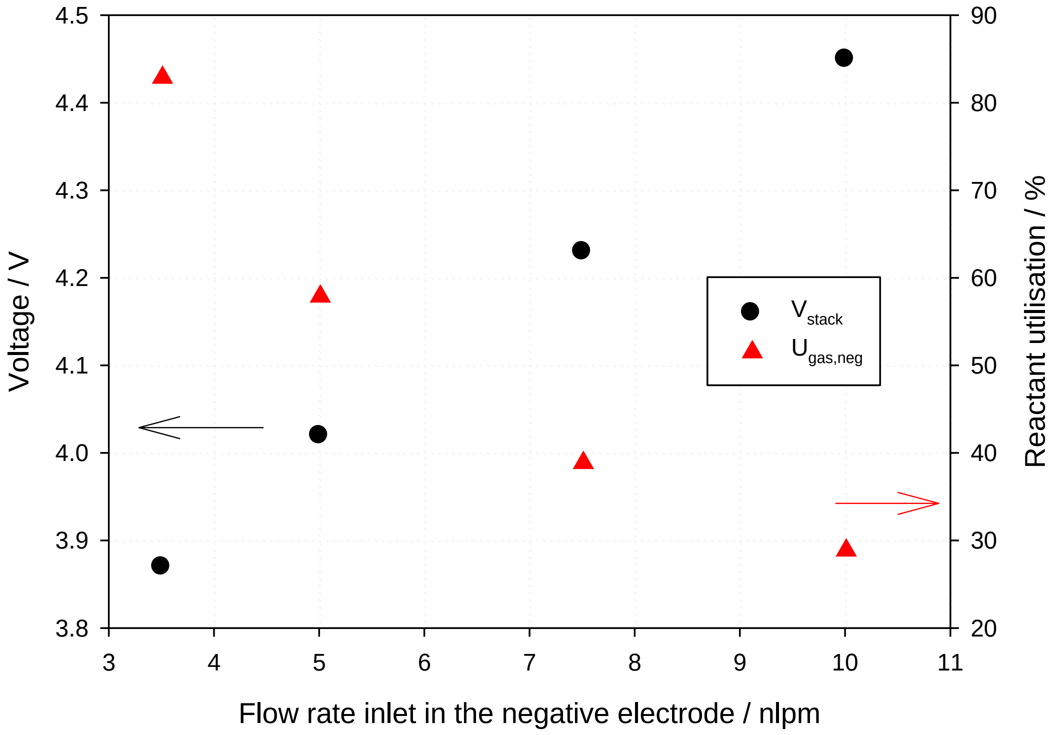


Figure 3: Example graph of representation of results in SOFC mode when varying $f_{neg,in}$ and keeping constant I (50 A), $x_{H2,neg,in}$ (0.6) and $f_{pos,in}$ (20 nlpm) in a 5-cell stack.

8 Differences to Existing Procedures

This TM regards operation of the samples in power generation (SOFC) and electrolysis mode (SOEC), while FCTES^{QA} and the IEC 62282-7-2 only take into consideration operation in SOFC mode. It must be noted that this TM is similar to the *Lambda Sensitivity* test module created in STACKTEST Project, aimed for polymer electrolyte fuel cells (PEMs).

9 Bibliography

[1] Test Module 2.06: Lambda Sensitivity", Document of EU-Project Stacktest, Version 4.1, 02.07.2014, http://stacktest.zsw-bw.de.

[2] Test Module TM00 "General SOC Testing Guidelines".

Test Module 08

Reactant Gas Composition

(Version 1.8, 10 May 2017)

Objective and Scope

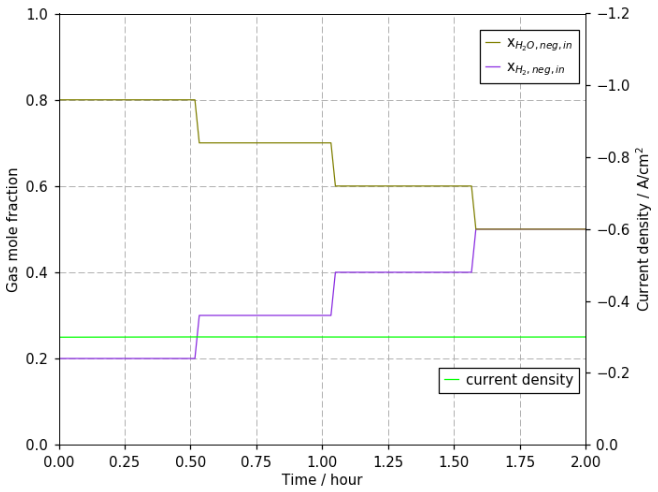
This test module deals with solid oxide cell (SOC) operation at different reactant composition either as a fuel cell (SOFC) or as an electrolyser (SOEC) to determine the influence of the reactant composition on the performance of an SOC cell/stack. It is a general characterization method that can be used in SOC R&D and for quality assurance.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Flow rates of inlet gases (f_{in})	Composition of inlet gases ($x_{i,\,in}$)
Temperature of the oven (T_{oven})	
Current (I)	

Test Procedure

- Set the stack temperature to the defined temperature. Change the reactant flows/compositions to the start point of the defined reactant gas flows/compositions.
- Change the stack/cell current to the defined current.
- Keep one of the electrode gas composition fixed while varying the reactant composition of the other electrode.



Example of evolution of inlet gas mole fraction during test (SOEC mode)

Critical Parameters and Parameter Controls

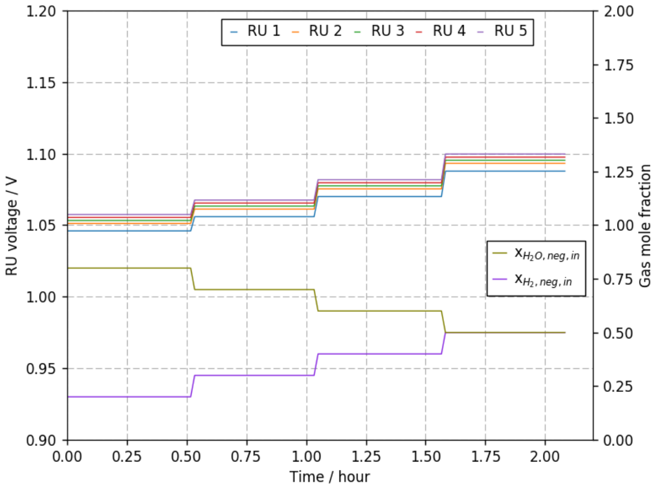
- Stability of stack/cell temperatures under each changing of gas composition.
- In SOEC mode, special care towards a stable supply of steam should be taken, so that the fluctuation at OCV for a single cell/RU is less than e.g., ±10 mV per cell.

Main Test Output Parameters (TOPs) and Derived Quantities

TOP	Derived Quantities
Voltage of cell/RU/stack (V)	Reactant gas utilization (U_{gas})
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	Electrical power density ($P_{d,el}$)

Data Post Processing and Representation

Representation examples of influence of changing of reactant composition:



Example of RU voltage evolution with changing of inlet gas composition at -0.3 A/cm² in SOEC mode

SOCTESQA:
Solid Oxide Cell and Stack Testing, Safety and Quality Assurance
Project website: www.soctesqa.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

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

THEME [SP1-JTI-FCH.2013.5.4]

Start date*: 01.05.2014 –* ***Duration****: 36 months*

Project Coordinator*: M. Lang – DLR*

Test Module 08: Reactant Gas Composition

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Version: 1.8

Last revision: 10.05.2017

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):

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

gas utilization at current I:

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

The average RU voltage as:

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

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

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

The average stack temperature as:

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

The electrical power as:

$$P_{el} = V_{stack} \times I \tag{7}$$

(Area specific) electrical power density as:

$$P_{d,el} = \frac{P_{el}}{A \times N} \tag{8}$$

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, $\mathrm{f}_{\mathrm{i,neg,in}}$ 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.

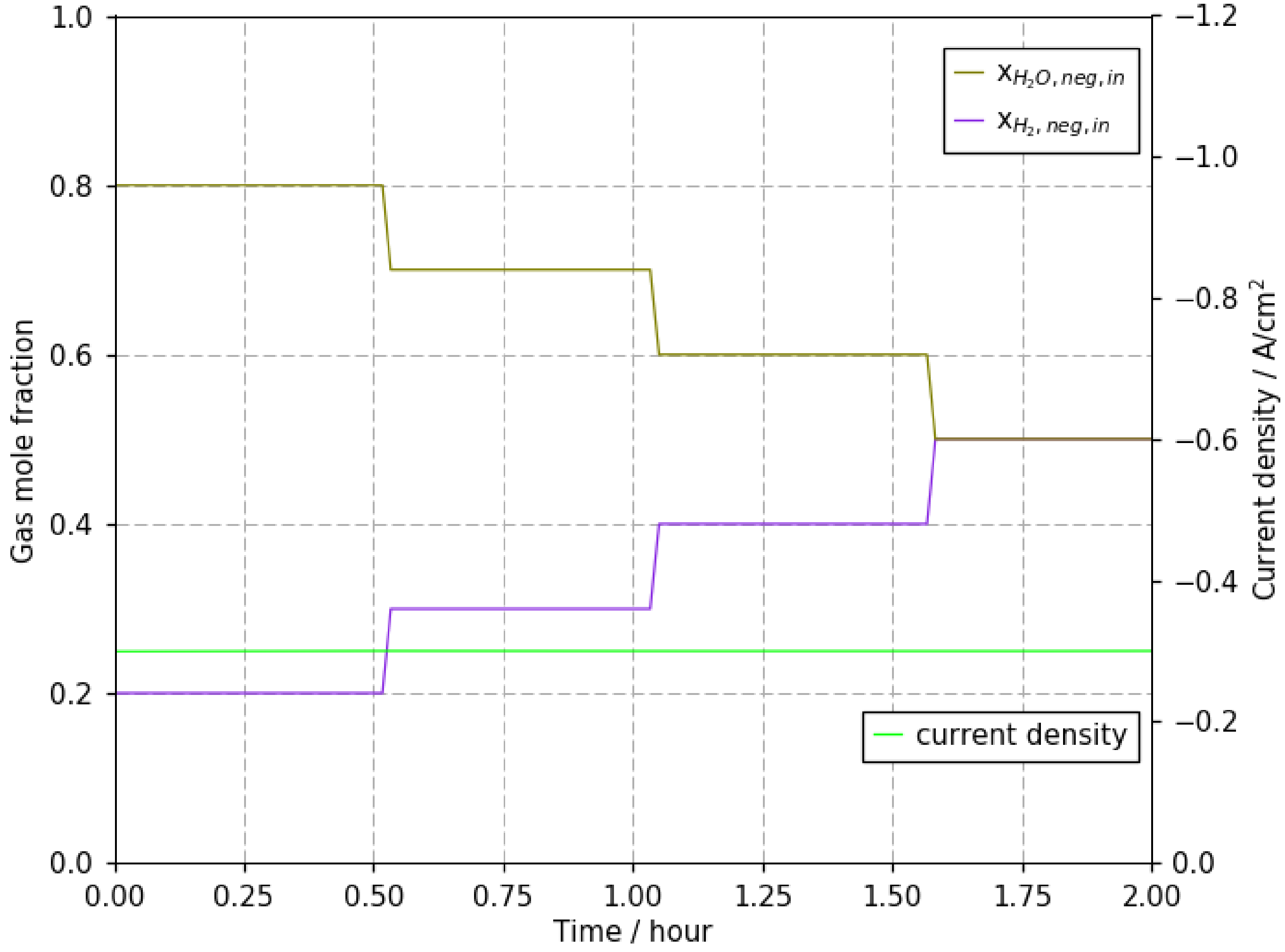


Figure 1: Example of inlet gas composition and current density as function of time in SOEC mode

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.

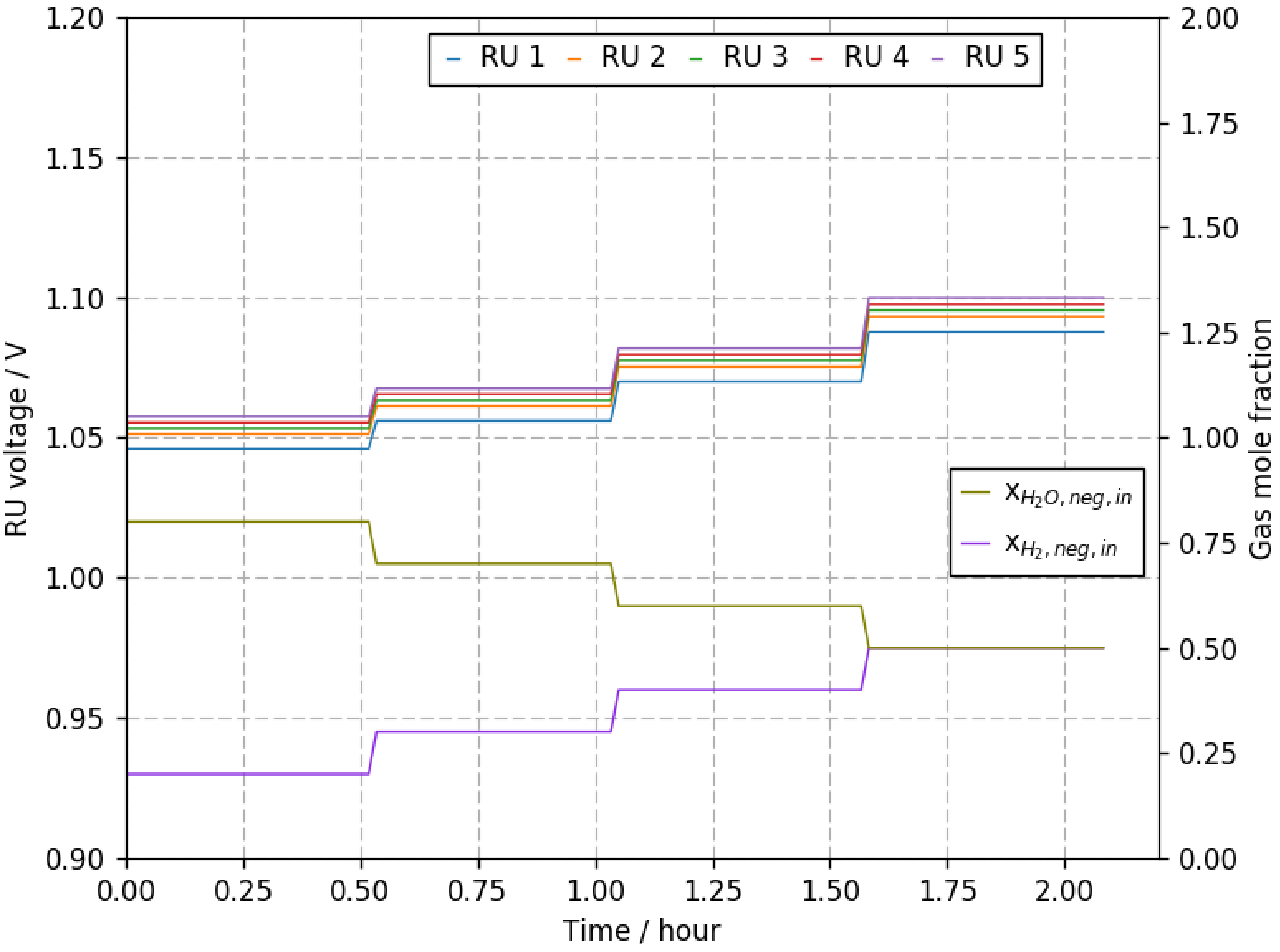


Figure 2: Example of RU voltage evolution with changing of inlet gas composition at -0.3 A/cm^2 in SOEC mode

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

1. Technical specification: “Single cell and stack test methods – Single cell and stack performance tests for solid oxide fuel cells”, International Electrotechnical Commission, IEC TC 105, IEC 62282-7-2, Part 7-2, 2010, to be published under http://webstore.iec.ch.
2. TM_P-05_Fuel_Oxidant_Composition, EU-Project Stacktest, Version 4.1, 02.07.2014, http://stacktest.zsw-bw.de.

Test Module 09

Temperature Sensitivity

(Version 6.2, 9 May 2017)

Objective and Scope

The purpose of this test module is to investigate the influence of the cell/stack nominal operating temperature on the cell/stack performance. The parameters that have a predominant role in this test module are: stack environment temperature (oven and pre-heaters) and current.

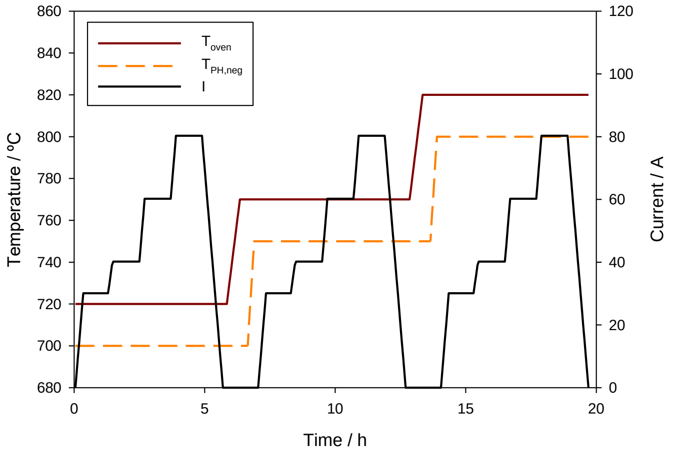
This test procedure has no specific target application. However, this test module describes a general characterization method that can be used in research and development of the SOC and for quality assurance in cell and stack production.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Flow rates of inlet gases (f_{in})	Current (I)
Composition of inlet gases ($x_{i,\,in}$)	Temperature of the oven (T_{oven})
Pressure of outlet gases (p_{out})	Temperature of pre-heater (T_{PH})

Test Procedure

The cell/stack temperature ranges under investigation, the current load levels, the number of individual test steps and the test operating conditions, have to be defined before the test. The tested cell/stack temperatures have to be chosen considering the allowable operating conditions specified by the cell/stack manufacturer. Based on these parameters the test point matrix can be prepared.



Example of test procedure

Critical Parameters and Parameter Controls

It is recommended to measure the cell/stack temperature at a fixed location which should be as close as possible to the geometric centre of the cell/stack. Since usually it will not be possible to place a thermocouple (or similar) in the heart of a stack, it is suggested to measure the temperature in direct contact with the centre of the end plate(s) of the cell/stack assembly. For stack tests, if possible, it is recommended to use two temperature sensors, one at the top and one at the bottom end plate and take the arithmetic average of the two values. If direct measurement of the cell/stack temperature is not possible, the oxidant outlet temperature should be taken as reference, measured as close as possible to the cell/stack outlet.

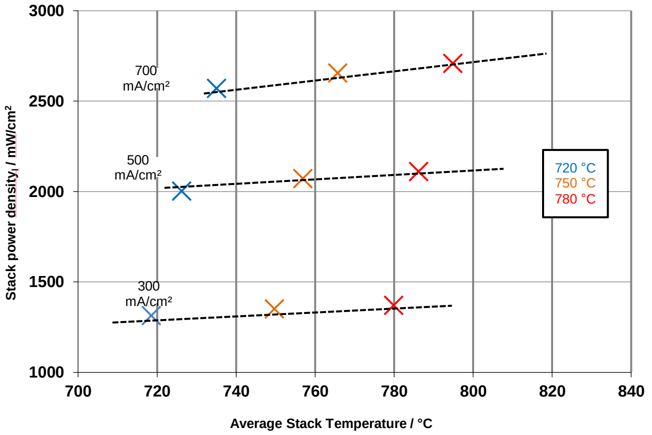
Changes in the cell/stack temperature caused by electrical load variation have to be considered. However, compensating changes in the cell/stack temperature caused by electrical load variation may be unfeasible, so that the *nominal* cell/stack temperature shall be taken at a given current density (either OCV or a nominal current density).

Main Test Output Parameters (TOPs) and Derived Quantities

TOP	Derived Quantities
Voltage of cell/RU/stack (V)	Current density (j)
Temperature of gas streams at cell/stack inlet/outlet (T)	Electrical power density ($P_{d,el}$)
Temperature of cell/stack (T)	Pressure drop of gas stream (ΔP_{gas})

Data Post Processing and Representation

The figure below is an example of how to correlate graphically the TIPs and TOPs of this test module. In this particular example graph represents the results to an analogous case, where the current and the oven temperature are the variable TIPs, while $f_{neg/pos,in}$ are static TIPs. More particularly, the three temperatures studied were $T_{cell/stack}$ = 720, 750 and 780ºC; the current densities requested to the short stack were: 300, 500 and 700mA/cm².



Temperature trend during temperature sensitivity

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU GRANT AGREEMENT N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
Start date*: 01.05.2014 –* ***Duration****: 36 months*
Project Coordinator*: M. Lang – DLR*

Test Module 09: Temperature Sensitivity

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Content

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 09 – Temperature Sensitivity

1 Objective and Scope

The purpose of this test module (TM) is to investigate the influence of the cell/stack nominal operating temperature on the cell/stack performance. The parameters that have a predominant role in this TM are: stack environment temperature (oven and pre-heaters) and current.

This test procedure has no specific target application. However, this test module describes a general characterization method that can be used in research and development of the SOC and for quality assurance in cell and stack production. This module is applied in combination with other test modules in a test programme. The test programme will be application-specific and also describe the operating conditions of the SOC in more detail. All the quantities used in TM09 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.

Note: the parameters, values and range of values including uncertainties used throughout this document are recommended only.

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)

There are two types of test input parameters (TIPs): variable TIPs and static TIPs (for definitions refer to Table 1 in TM00).

The measurement uncertainties, the sampling rates and the values given in the following tables are in compliance with the master document TM00. Deviations from the test module should be reported in the test report.

Table 1 lists the static TIPs for the temperature sensitivity analysis when the effect needs to be assessed of a variation different nominal operating temperature levels on cell/stack performance. The variable TIPs for this test are listed in Table 2.

Table 1: Static test input parameters TIPs in TM09 for SOFC and SOEC modes as defined in TM00

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
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}
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	$x_{i,pos,in}$	-	-
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)
Rate of oven temperature change	$\Delta T_{oven}/\Delta t$	K min^{-1}	K s^{-1}
Rate of pre-heater temperature change	$\Delta T_{PH}/\Delta t$	K min^{-1}	K s^{-1}

Table 2: Variable test input parameters TIPs in TM09 for SOFC and SOEC modes as defined in TM00

Description of quantity	**Symbol**	**Unit often used**	**SI Unit**
Electrical current through the cell/stack	I	A	A
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

4 Test Output Parameters (TOPs)

The main output parameters are the average cell/stack temperature and voltages of the stack and RUs. These test outputs have to be recorded at the targeted cell/stack nominal operating temperature levels, while varying *I*, or the stack environment temperatures, $T_{PH,neg/pos}$ *and* T_{oven}. There can be many TOPs attending to the availability of apposite measurement/recording devices yet, Table 3 illustrates only the recommended parameters for this Test Module.

Table 3: Test output parameters TOPs in TM09 for SOFC and SOEC modes as defined in TM00

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
Voltage of the stack	V_{stack}	V	V
Voltage of repeating unit (RU) i in the stack	$V_{RU,i}$	V	V
Voltage of the cell	V_{cell}	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
Temperature of the bottom plate of the stack	T_{BP}	°C	K
Temperature of the cell	T_{cell}	°C	K
Stack temperature	T_{stack}	°C	K
Temperature of the top plate of the stack	T_{TP}	°C	K

5 Derived quantities

The parameters listed in Table 4 are important and commonly used values calculated from input and output parameters.

Table 4: Derived quantities in TM09 Temperature sensitivity as defined in TM00

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
Electrical current density through the cell/stack	j	A cm^{-2}	A m^{-2}
Electrical power of the cell/stack	P_{el}	W	J s^{-1} (W)
Pressure drop of the negative electrode gas stream over the cell/stack	Δp_{neg}	mbar, kPa	N m^{-2} (Pa)
Average temperature of the stack	T_{av}	ºC	K
Maximum temperature difference in the stack	ΔT_{max}	ºC	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, for example, as follows:

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

A stack can be damaged during the temperature ramps if the temperature gradient between the gas inlets and the stack itself is too high. A value for the maximum temperature difference during temperature ramps 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

6.1 Critical Parameters and Parameter Controls

6.1.1 Sensor position

It is recommended to measure the cell/stack temperature at a fixed location which should be as close as possible to the geometric centre of the cell/stack. Since usually it will not be possible to place a thermocouple (or similar) in the heart of a stack, it is suggested to measure the temperature in direct contact with the centre of the end plate(s) of the cell/stack assembly. For stack tests, if possible, it is recommended to use two temperature sensors, one at the top and one at the bottom end plate and take the arithmetic average of the two values. If direct measurement of the cell/stack temperature is not possible, the oxidant outlet temperature should be taken as reference, measured as close as possible to the cell/stack outlet.

6.1.2 Temperature stability

The reproducibility of the cell/stack performance with temperature during the measurement time has to be assured. Minor temperature variations are acceptable as long as the cell/stack voltage is not influenced significantly. Since the objective of the test is to determine the influence of cell/stack nominal operating temperature on cell/stack performance, changes in the cell/stack temperature caused by electrical load variation have to be considered. However, compensating changes in the cell/stack temperature caused by electrical load variation may be unfeasible, so that the *nominal* cell/stack temperature shall be taken at a given current density (either OCV or a nominal current density). Afterwards the *effective* cell/stack temperature at each test step, after the defined stabilisation time (see section 6.4 of TM00) and at which the corresponding cell/stack voltage shall be taken, shall be noted in the test output table.

At each test step, the cell/stack assembly unit shall be operated until it reaches the stable state in terms of temperature and voltage (stabilisation time). At stable state, measurements are taken for the entire duration of the prescribed sampling interval (measurement time). The average value of these measurements shall be the measured value for that step.

6.2 Preconditioning of the Stack

The preconditioning of the fuel cell stack is specified in the SOCTESQA master document and should be realized under nominal conditions given by the stack manufacturer.

6.3 Carrying out the Test for Temperature Sensitivity

6.3.1 Test Methodology

The cell/stack temperature ranges under investigation, the current load levels, the number of individual test steps and the test operating conditions, have to be defined before the test. The tested cell/stack temperatures have to be chosen considering the allowable operating conditions specified by the cell/stack manufacturer. Based on these parameters the test point matrix can be prepared.

Example

This test module can be used to determine the behaviour of a stack under 25, 50, 75 and 100% of the nominal load when the nominal operating temperature is varied by multiples of 50°C and $f_{neg/pos,in}$ remain constant. The resulting test point matrix is presented in Table 5.

Table 5: Test example

$T_{cell/stack}$ (ºC) @ I nominal	I (A)	$f_{neg,in}$ (nlpm)	$f_{pos,in}$ (nlpm)
700	**30.1**	10	20
700	**40.2**	10	20
700	**60.2**	10	20
700	**80.3**	10	20
750	**30.1**	10	20
750	**40.2**	10	20
750	**60.2**	10	20
750	**80.3**	10	20
800	**30.1**	10	20
800	**40.2**	10	20
800	**60.2**	10	20
800	**80.3**	10	20

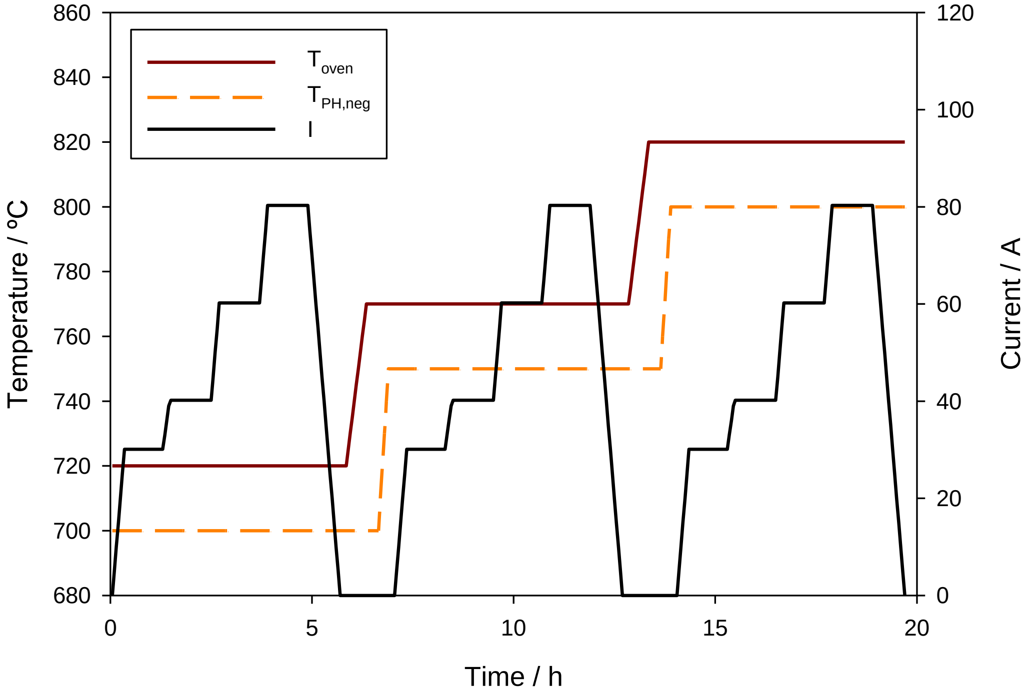


Figure 1: Qualitative representation of TIPs when carrying out TM09 using the values from Table 5.

6.3.2 Stabilization times

For stabilization times, please refer to the stack or cell manufacturer/supplier. In case a stabilization period is not provided, the procedure described in TM00 (Section 6.5) can be used.

The dwell-time (stabilisation time plus measurement time) for each test point is defined by the stability criteria of TIPs and TOPs (see TM00). Stabilisation time and measurement time will depend on the test objective. The following minimum time scale is recommended, but can be adjusted to the test objective to decrease the overall test duration:

- stabilisation time: 25 minutes
- measurement time: 5 minutes
- resulting dwell-time: 30 minutes

7 Data Post-processing and Representation

All of the TOPs can be represented in one single graph as a function of time. It is important to clearly define the cell/stack temperature that is maintained for results comparison, as the local temperatures in the cell/stack environment may vary significantly. This graph represents the results to an analogous case to that explained in the previous example, where the current and the oven temperature are the variable TIPs, while $f_{neg/pos,in}$ are static TIPs. More particularly, the three temperatures studied were $T_{cell/stack}$ = 720, 750 and 780ºC; the current densities requested to the short stack were: 300, 500 and 700 mA/cm^2.

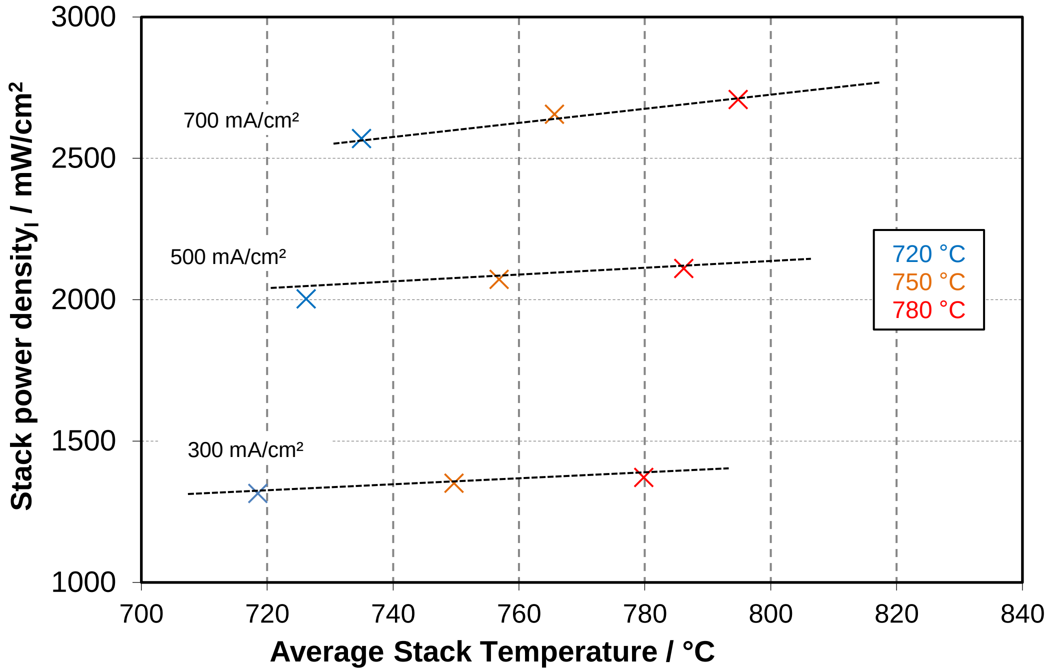


Figure 2: Temperature trends during the temperature sensitivity analysis when the current and the oven temperature are the variable TIPs.

8 Differences to Existing Procedures

No test procedures have been defined and elaborated in the international standardization literature specifically on the temperature sensitivity of a SOC cell/stack at the time of writing. This TM is particularly relevant as it can address the effects of temperature on the performance of the SOC when operating in power generation mode (SOFC) and electrolysis mode (SOEC). It must be noted that this TM is similar to the *Temperature Sensitivity* test module created in STACKTEST Project, aimed for polymer electrolyte fuel cells (PEMFCs).

9 Bibliography

[1] “Test Module 2.06: Temperature sensitivity”, Document of EU-Project Stacktest, Version 4.1, 02.07.2014, http://stacktest.zsw-bw.de.

[2] Test Module TM00 of SOCTESQA

Test Module 12
Operation under Constant Current

(Version 3.4, 5 May 2017)

Objective and Scope

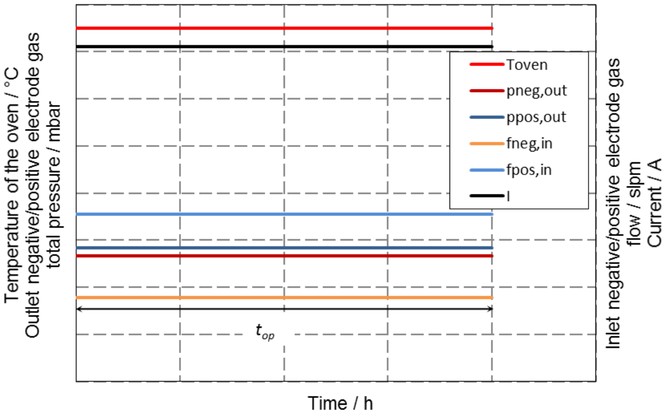
This test module deals with solid oxide cell (SOC) operation under constant current either as a fuel cell (SOFC) or as an electrolyser (SOEC). The aim is to establish a widely accepted method for performing a long-term steady-state endurance test. A calculation method of the SOC degradation rate is also recommended from the continuously recorded SOC voltages as a function of time. Together with data obtained from other test modules (TM03 “*Current-voltage characteristics*” and TM04 “*Electrochemical impedance spectroscopy*”) performed before and after the long-term steady-state operation, a comprehensive representation of the degradation behaviour of SOC can be achieved.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Current (I)	(None)
Flow rates of inlet gases (f_{in})	
Composition of inlet gases ($x_{i,\ in}$)	
Pressure of outlet gases (p_{out})	
Temperature of the oven (T_{oven})	

Test Procedure

- Set the operating conditions according to the predefined operating point and check the stability of static TIPs.
- Let operation last for a predefined duration. Record all relevant TIPs and TOPs during the operation.
- The test can be interrupted or terminated when operational abnormalities (such as unexpected temperature evolution, signal instabilities) are observed or certain predefined cut-off criteria are fulfilled (threshold values on voltage, temperature or degradation rate).



General evolution of TIPs during TM12

Critical Parameters and Parameter Controls

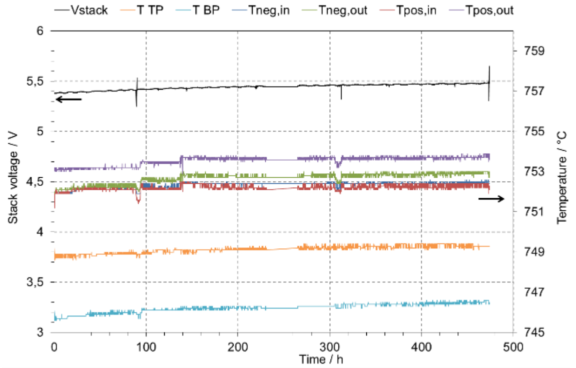
- Particular attention has to be paid to the stability of the operating conditions (temperature, pressure, gas flows and current).
- Voltage measurement as a function of time has to be sufficiently clean to allow degradation rate determination during long-term operation. In SOEC mode, special attention has to be paid to a stable supply of steam to minimize voltage fluctuation.

Main Test Output Parameters (TOPs) and Derived Quantities

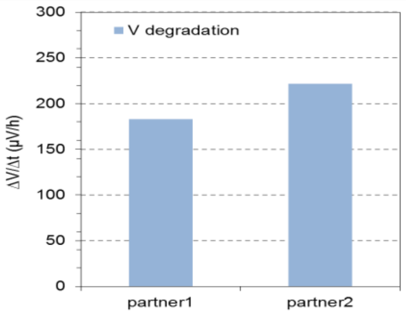
TOP	Derived Quantities
Voltage of cell/RU/stack (V)	Current density (j)
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	Degradation rate of cell/RU/stack voltage ($\Delta V/\Delta t$)
Flow rates of outlet gases (f_{out})	Reactant gas utilization (U_{gas})
Composition of outlet gases ($x_{i,\ out}$)	Average temperature of the stack (T_{av})

Data Post Processing and Representation

Examples of data representation in SOEC mode during TM12:



Evolution of stack voltage and temperatures with time for a steady-state operation at a constant current density of -0.3 A cm^{-2}.



Calculated voltage degradation rates between two test partners.

SOCTESQA:
Solid Oxide Cell and Stack Testing, Safety and Quality Assurance
Project website: www.soctesqa.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU Grant Agreement N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
***Start date**: 01.05.2014 – **Duration**: 36 months*
***Project Coordinator**: M. Lang – DLR*

Test Module 12: Operation under Constant Current

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Content

Abbreviations

APU	Auxiliary power unit
ASR	Area specific resistance
CHP	Combined heat and power
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 12 – Operation under Constant Current

1 Objective and Scope

This document presents the test module 12 (TM12) which deals with solid oxide cell (SOC) operation under constant current (i.e. galvanostatic conditions) either as a fuel cell (SOFC) or as an electrolyser (SOEC). The aim of this test module is to establish a widely accepted method for SOC steady-state operation when a long-term endurance test is performed. A calculation method of the SOC degradation rate is also recommended from the continuous SOC voltages recording as a function of time. The particular interest of this method consists in achieving the voltage evolution/dependency over time and so to know if SOC performance evolves steadily or not. Other TMs, such as TM03 “Current-voltage characteristics” and TM04 “Electrochemical impedance spectroscopy”, also allow to calculate SOC degradation rates of cell/stack resistances (ohmic and polarization). It is necessary to provide different representations of degradation in order to give a comprehensive representation of the cell/stack assembly unit durability.

This test module addresses SOC cell/stack assembly units, testing systems, instruments, measuring methods and test methods. Moreover, it is also applicable to the four different SOC applications selected in the SOCTESQA project:

- Stationary and distributed power generation (SOFC-µCHP)
- Mobile (SOFC-APU)
- H_2 production in power-to-gas (SOEC)
- Electricity storage in power-to-gas-to-power (SOEC/SOFC)

Definition of relevant steady-state operating conditions related to these applications are supported by recommendations of the SOCTESQA Industrial Advisory Board members. This test module follows the recommendations of the IEC international standard [1] when applicable. All the quantities used in TM12 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)

There are two types of test input parameters: variable and static. The first type may vary during the duration of the TM while the second type does not vary during the overall duration of the TM. TM12 doesn’t include any variable TIPs. The SOC assembly units are usually operated in galvanostatic mode (at a constant current for a given operating point).

TM12 starts by setting the following parameters as constants: oven temperature, total pressure (atmospheric or higher) as well as gas flow rates at the negative and positive electrodes and finally the magnitude of the current after a period of stabilization of the first parameters. Those parameters are chosen according to full operating conditions i.e. in relation to relevant initial voltages and gas utilizations at the negative and positive electrodes (fuel utilization (SOFC) or steam conversion rate (SOEC) at the negative electrode and air / oxygen utilization (SOFC) at the positive electrode) for each application.

Table 1 below lists all the relevant test input parameters (operating conditions) useful in TM12. They all belong to the static category. T_{oven} (as well as $T_{PH,neg}$ *and* $T_{PH,pos}$ when available) is controlled and kept constant all along the TM according to the targeted initial operating T_{cell} or T_{stack} (at OCV before increasing current). Optionally, it is also possible to fix T_{oven} under polarization. $T_{neg,in}$ and $T_{pos,in}$ can vary as well as T_{cell} or T_{stack} during the TM. The following pressure values $p_{neg,out}$, and $p_{pos,out}$ must also be controlled so as to obtain the targeted value at the inlet in relation to the measured pressure drop over the cell/stack assembly unit (Δp_{neg}, Δp_{pos}). $f_{neg,in}$, $f_{pos,in}$, $f_{i,neg,in}$ and $f_{i,pos,in}$ gas flows are also controlled to fix the mole fractions of gas components $x_{i,neg,in}$ and $x_{i,pos,in}$ and in association with the current I, to achieve the targeted gas utilizations at both electrodes ($U_{gas,neg}$ and $U_{gas,pos}$) and the initial voltage as well (initial values of V_{stack}, V_{cell} and $V_{RU,av}$). The absolute current $|I|$ is maintained to the targeted value for a certain operating time t_{op} [2-5].

Table 1: Static test input parameters during TM12.

Description of quantity	**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}
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}
Electrical current through the cell/stack	I	A	A
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)
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
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 2 below shows the list of the test output parameters that can be recorded during the overall duration of TM12, mainly the cell/stack assembly unit response e.g. V_{stack}, V_{cell} and $V_{RU,i}$ SOC voltages [2,3,4]. In association with the current I and for long-term endurance tests, those TOPs allow the calculation of the SOC cell/stack performance degradation rates on voltage as described in the master document TM00 (section 10).

T_{cell}, T_{stack}, T_{TP}, T_{BP}, $T_{neg,in}$, $T_{pos,in}$, $T_{neg,out}$ 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 2: Test output parameters during TM12.

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 the repeating unit (RU) *i* in the stack	$V_{RU,i}$	V	V
Temperature of the cell	T_{cell}	°C	K
Temperature of the stack	T_{stack}	°C	K
Temperature of the top plate	T_{TP}	°C	K
Temperature of the bottom plate	T_{BP}	°C	K
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
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}	$m^3\ s^{-1}$
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}	$m^3\ s^{-1}$
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

The following Table 3 gives the derived quantities useful for this TM [1,2]. They are all calculated from TIPs and TOPs with the equations presented in TM00 - section 10.

Table 3: Derived quantities possibly calculated during TM12.

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
Electrical current density through the cell/stack	j	A cm^{-2}	A m^{-2}
Gas utilization at the negative electrode	$U_{gas,neg}$	-	-
Gas utilization at the positive electrode	$U_{gas,pos}$	-	-
Average RU voltage of all RUs in the stack	$V_{RU,av}$	V	V
Average temperature of the stack	T_{av}	°C	K

Degradation rate of cell voltage	$\Delta V_{cell}/\Delta t$	V s^{-1}	V s^{-1}
Degradation rate of stack voltage	$\Delta V_{stack}/\Delta t$	V s^{-1}	V s^{-1}
Degradation rate of repeating unit (RU) *i* voltage	$\Delta V_{RU,i}/\Delta t$	V s^{-1}	V s^{-1}

For long-term endurance test, the degradation rate is reported as the change in voltage (ΔV_{cell}, ΔV_{stack} and $\Delta V_{RU,i}$) over a given time $(t_1\text{-}t_0)$ which has to be clearly mentioned (see TM00 – section 10). Indeed, for instance, the first hundred hours are generally disregarded ($(t_1\text{-}t_0) < t_{op}$) because the cell/stack assembly unit follows a kind of transient operation and the degradation rate can be very high in this period. This degradation rate is expressed in mV per kh (or µV per h), as defined in the section 10 of the master document TM00 and reference [2]. It can additionally be expressed in % of the initial value.

The absolute degradation ΔX of a quantity X within the time from t_0 to t_1 is calculated as the difference between the final value $X(t_1)$ and the initial value $X(t_0)$:

$$\Delta X = X(t_1) - X(t_0)$$

The relative degradation ΔX_{rel} is calculated by dividing ΔX by the initial value $X(t_0)$:

$$\Delta X_{rel} = \frac{X(t_1) - X(t_0)}{X(t_0)} \cdot 100\%$$

The degradation rate (rate of change) of quantity X during the time interval $(t_1\text{-}t_0)$ is then calculated by:

$\frac{\Delta X}{\Delta t} = \frac{\Delta X}{t_1 - t_0}$ with the unit [unit of *X*/time unit]

$\frac{\Delta X_{rel}}{\Delta t} = \frac{\Delta X_{rel}}{t_1 - t_0}$ with the unit [%/time unit]

Degradation rates are typically expressed by the absolute or relative change per 1000 hours. It is thus advisable to normalize the results to 1000 h time interval. This can simply done by converting the unit of time interval to kh.

6 Test Procedure

This specific TM12 is dedicated to SOC steady-state operation for long-term endurance tests. The aim of this TM consists in recording performance evolution (i.e. cell/stack voltage in galvanostatic mode) over a period of time at constant nominal operating conditions (temperature, pressure, gas flows and current) dependent on each application.

6.1 Critical parameters and parameter controls

In this specific TM, particular attention has to be paid to the stability of the operating conditions (temperature, pressure, gas flows and current) when constant. Also voltage measurement as a function of time has to be sufficiently clean to allow degradation rate determination during long-term operation.

As mentioned in D5.3, the importance of avoiding pollution from the test bench and inlet gases can be pointed out here, since it has a strong influence on the degradation.

6.2 Operation under constant current

Realistic operating points have to be defined for each selected application (stationary SOFC-µCHP and distributed power generation, mobile SOFC-APU application, SOEC for H_2 production (power-to-gas) and combined SOFC/SOEC for energy storage (power-to-gas-to-power) for instance) in terms of targeted initial V_{cell}, V_{stack} and $V_{RU,av}$ voltages as well as $U_{gas,neg}$ and $U_{gas,pos}$ gas utilizations. At relevant temperatures, inlet gases compositions and flow rates for each application, a realistic current I and operation duration t_{op} have to be defined based on literature and manufacturers recommendations.

Figure 1 below presents the general TIPs' evolution all along this TM.

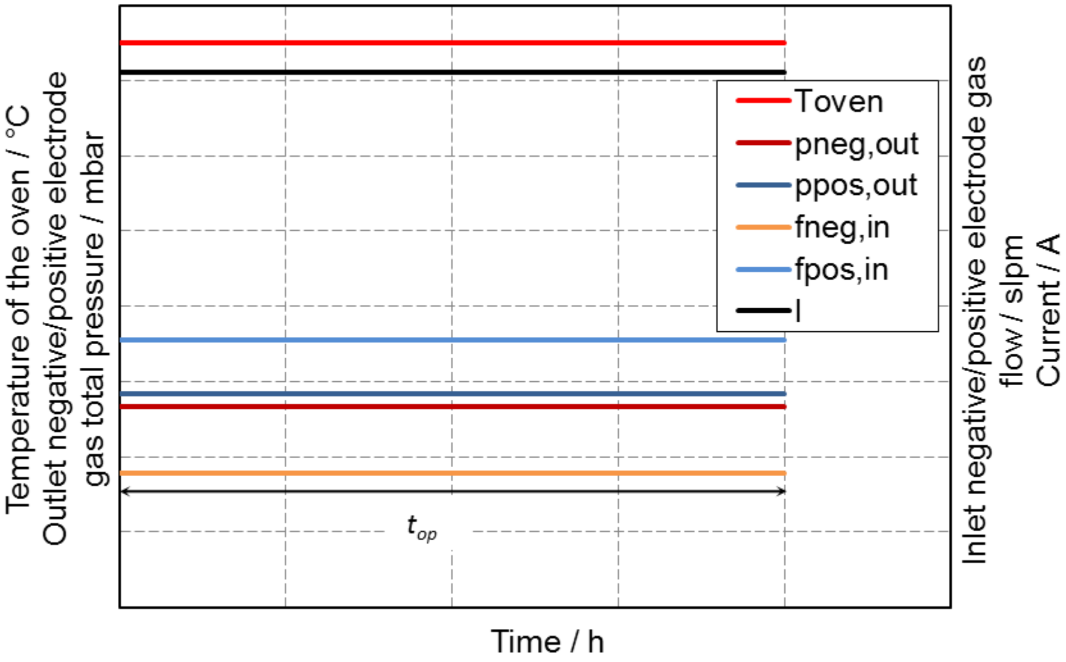


Figure 1: General evolution of TIPs during TM12.

1) Check that the stability of the static TIPs (T_{oven}, $p_{neg,out}$, $p_{pos,out}$, $f_{neg,in}$, $f_{pos,in}$, $f_{i,neg,in}$, $f_{i,pos,in}$ and I) is acceptable;

2) Let operation last for t_{op}. It is recommended to measure and record properly all TIPs and TOPs mentioned in sections 3 and 4. In particular, the evolution of the V_{cell}, V_{stack} and $V_{RU,i}$ voltages and of the T_{cell} or T_{stack} is carefully recorded as a function of time during the overall duration of TM12. If a leak is suspected at any moment during the test (unexpected temperature evolution, signal instabilities), stack operation can be interrupted by decreasing the current absolute value to zero and OCV value checked. In any operational mode (SOFC, SOEC), an automatic cut-off voltage threshold may also be set, bringing immediately to stop the current density even if the foreseen duration t_{op} is not achieved yet. Alternatively, a value of voltage degradation (decrease in SOFC mode and increase in SOEC mode) can be defined to stop the test module automatically even if the foreseen duration t_{op} is not achieved yet. An automatic cut-off criteria may also be chosen on T_{cell} or T_{stack}.

7 Data Post Processing and Representation

Information on reporting of test results is mentioned in the section 9 of the master document TM00. In particular, for this TM, a graph presenting the evolution of V_{cell}, V_{stack} and $V_{RU,i}$ voltages as a function of time is highly recommended as well as a graph with the time-evolution of T_{cell} or T_{stack} (see *Figure 2* for stack voltage and temperatures evolution in SOEC mode for instance).

Also the calculated degradation rate of the SOC voltage can be presented in a graph as shown in *Figure 3*. If a strong temperature evolution is observed during the operation under constant current, the determination of the degradation rate will be largely influenced. In this case, a temperature correction can be applied:

- either by adapting the oven temperature at the end of the TM12 step in order to bring the stack temperature back to the initial value and take the obtained voltage as the final one,

- or by performing the TM12 step at a constant T_{stack} through constant adjustment of the oven temperature which can be automatically realized through the temperature regulation unit.

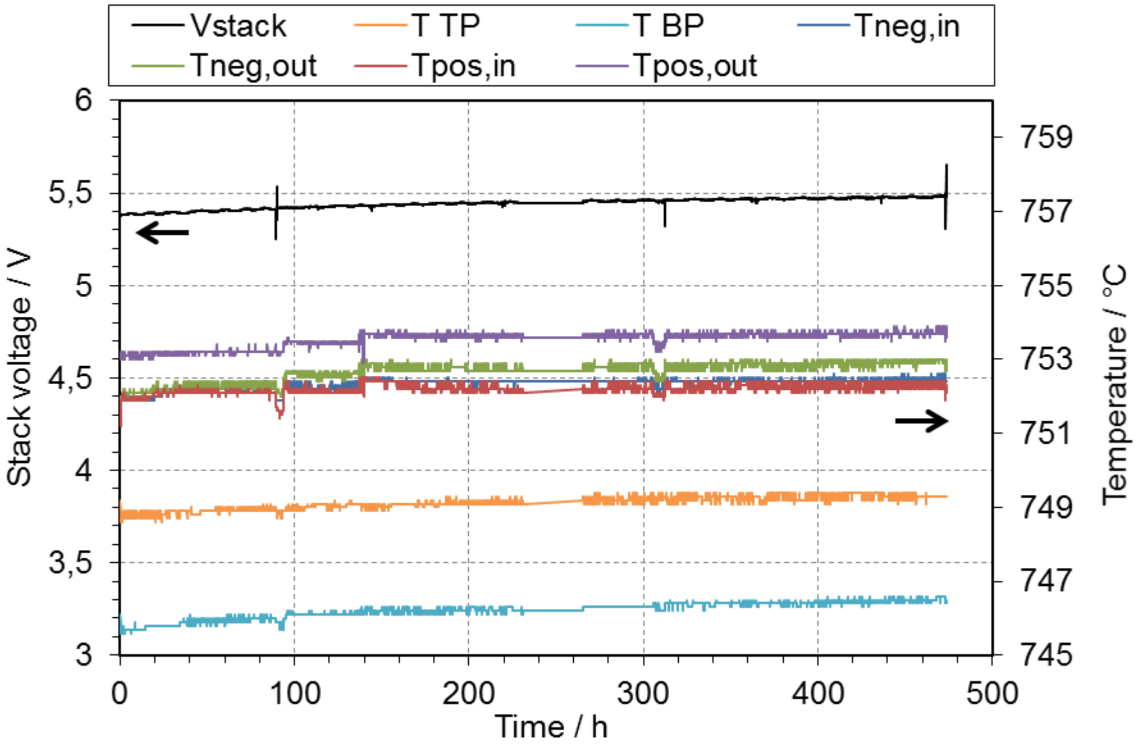


Figure 2: Example of data representation in SOEC mode during TM12: stack voltage and temperatures as a function of time for a constant current of -0.3 A/cm². Temperatures variation of about 1°C in this example.

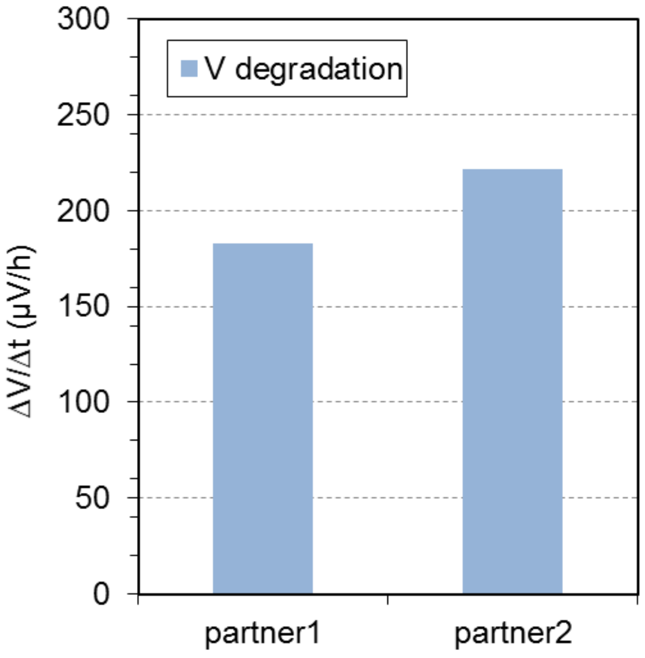


Figure 3: Example of data representation in SOEC mode during TM12: calculated voltage degradation rates between two partners.

8 Differences to Existing Procedures

This TM topic is quite common nowadays as shown by references found in literature [2-5]. Nevertheless, existing procedures, as reference [1], remain quite generic. Based on those references, the present TM12 fully dedicated to operation under constant current presents in details the relevant TIPs, TOPs and derived quantities with their associated formularies, their evolutions, the test procedure to perform long-term operation in both SOFC and SOEC modes and the different ways to express degradation rates in order to achieve a more comprehensive representation of the cell/stack assembly unit durability.

9 Bibliography

[1] 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)

[2] M. Näslund, H. Iskov, Delrapport DGC1: Accelerated lifetime testing and standardization of SOFC systems, Danish Gas Technology Centre, Horsholm 2012

[3] SOFC single cell performance and endurance test modules, Test Modules TM SOFC 01-04LD / 05-08HD, FCTESQA project, 2010.

[4] SOFC stacks performance and endurance test modules, Test Modules TM SOFC 25-28HD, FCTESQA project, 2010.

[5] ADEL deliverable D1.5 – Definition of ADEL testing protocol and revised test matrix, Q. Fu, A. Brisse, Eifer, 2013

Test Module 13

Operation under Varying Current

(Version 3.4, 11 May 2017)

Objective and Scope

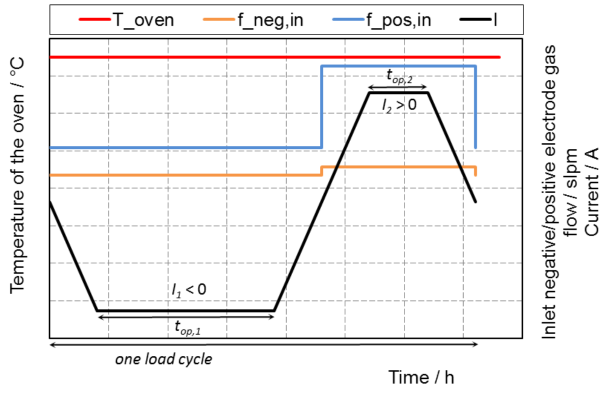
This test module deals with solid oxide cell (SOC) operation either as a fuel cell (SOFC) or an electrolyser (SOEC) under varying current under galvanostatic conditions to determine performance at relevant (intermittent) load profiles. It is a general characterization method that can be used in SOC R&D and for quality assurance.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Temperature of the oven (T_{oven})	Flow rates of inlet gases (f_{in})
Rate of change of current ($\Delta I/\Delta t$)	Current (I)
Operating time at the plateau d ($t_{op,d}$)	Composition of inlet gases ($x_{i,in}$)
Number of cycles and plateaus (m and d)	

Test Procedure

- Increase current from zero (open circuit voltage) to 1st plateau value of the current at its specified rate of change.
- Wait for $t_{op,1}$ to elapse and continuously record all TIPs & TOPs at their specified sampling rates, e.g. 1 Hz.
- Continue the current change/current holding until the dth plateau for $t_{op,d}$ to finish one cycle. Repeat until the number m of cycles is exhausted upon which the current is lowered to zero at its specified rate of change.
- The test can be interrupted or terminated when operational abnormalities (such as unexpected temperature evolution, signal instabilities) are observed or certain predefined cut-off criteria are fulfilled (threshold values on voltage, temperature or degradation rate).



General evolution of TIPs during TM13 in combined SOFC/SOFC operation for instance.

Critical Parameters and Parameter Controls

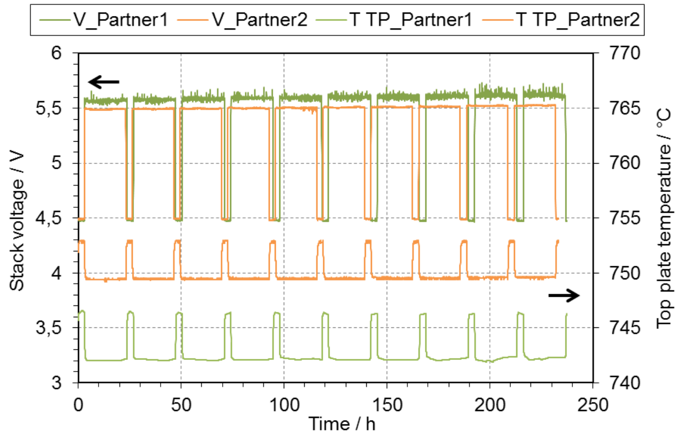
- Stability of T_{stack} and f_{in} at each load cycle plateau.
- Voltage measurement as a function of time has to be sufficiently clean to allow degradation rate determination during long-term operation. In SOEC mode special attention has to be paid to a stable steam supply to minimize voltage fluctuations.
- It is important to avoid pollution from inlet gases and the test bench itself, since it has a strong influence on the degradation.

Main Test Output Parameters (TOPs) and Derived Quantities

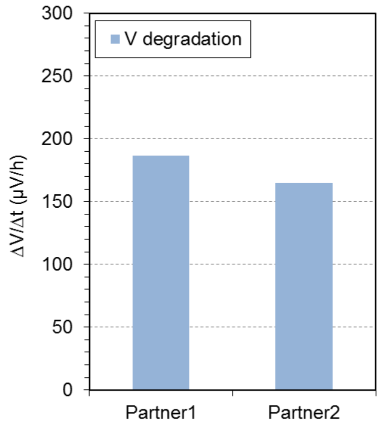
TOP	Derived Quantity
Voltage of cell/stack (V)	Current density (j)
Flow rates of outlet gases (f_{out})	Gas utilization (U_{gas})
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	Degradation rate of cell/stack voltage ($\Delta V/\Delta t$)
Composition of outlet gases ($x_{i,out}$)	Average temperature (T_{av})

Data Post Processing and Representation

Data representation examples under variable load in SOEC mode:



Evolution of SOEC stack voltage and temperature for load cycling between 0 and -0.3 A cm².



Calculated voltage degradation rates between two partners

SOCTESQA:
Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Project website: www.soctesqa.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU GRANT AGREEMENT N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
***Start date**: 01.05.2014 – **Duration**: 36 months*
***Project Coordinator**: M. Lang – DLR*

Test Module 13: Operation under Varying Current

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Content

Abbreviations

APU	Auxiliary power unit
ASR	Area specific resistance
CHP	Combined heat and power
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 13 – Operation under Varying Current

1 Objective and Scope

Test Module 13 (TM13) deals with solid oxide cell (SOC) operation under varying current when in galvanostatic conditions. The present document is valid for both fuel cell (SOFC) and electrolyser (SOEC) modes of the cell/stack. SOC assembly units are expected to operate under various loads depending on the electrical, thermal and/or gas demands. Besides the direct coupling with power generators relying on renewable energy sources might generate intermittent load patterns. The aim of this test module is to establish a widely accepted method for SOC dynamic operation under relevant load profiles. A calculation method of the SOC degradation rate is also recommended from the continuous SOC voltages recording as a function of time. The particular interest of this method consists in achieving the voltage evolution/dependency over time and so to know if SOC performance evolves steadily or not. Other TMs, such as TM03 "Current-voltage characteristics" and TM04 "Electrochemical impedance spectroscopy", also allow to calculate SOC degradation rates on resistances in particular. It is necessary to provide different representations of degradation in order to give a comprehensive representation of the cell/stack assembly unit durability.

This test module addresses SOC cell/stack assembly units, testing systems, instruments and measuring and test methods. Moreover, it is also applicable to the four SOC applications selected in the SOCTESQA project, which will strongly influence the load patterns to be tested:

- Stationary and distributed power generation (SOFC-µCHP),
- Mobile (SOFC-APU),
- H_2 production in power-to-gas (SOEC),
- Electricity storage in power-to-gas-to-power (SOEC/SOFC).

Definition of relevant load patterns related to these applications are supported by recommendations of the SOCTESQA Industrial Advisory Board members. This test module follows the recommendations of the IEC international standard [1] when applicable. All the quantities used in TM13 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 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)

There are two types of test input parameters: variable and static. The first type may vary during the duration of the TM while the second type does not vary during the overall duration of the TM. The SOC assembly units are usually operated in galvanostatic mode (at a constant current for a given operating point).

Table 1 and *Table 2* below list all the relevant test input parameters (operating conditions) useful in TM13. T_{oven} (as well as $T_{PH,neg}$ *and* $T_{PH,pos}$ when available) is controlled and kept constant all along the TM according to the targeted initial operating T_{cell} or T_{stack} (either at OCV before increasing current or at the desired operating current at a chosen current plateau). $T_{neg,in}$ and $T_{pos,in}$ can vary as well as T_{cell} or T_{stack} during the TM. The following pressure values $p_{neg,out}$, and $p_{pos,out}$ must also be controlled so as to obtain the targeted value at the inlet in relation to the measured pressure drop over the cell/stack assembly unit (Δp_{neg}, Δp_{pos}). $f_{neg,in}$, $f_{pos,in}$, $f_{i,neg,in}$ and $f_{i,pos,in}$ gas flows are also controlled to fix the mole fractions of gas components $x_{i,neg,in}$ and $x_{i,pos,in}$ in each chosen operating mode and in association with the current I, to achieve the targeted gas utilizations at both electrodes ($U_{gas,neg}$ and $U_{gas,pos}$) and the initial voltage as well (initial values of V_{cell}, V_{stack} and $V_{RU,av}$). Then gas flows, mole fractions and pressures are variable TIPs depending on the tested operating mode (SOFC, SOEC or combined SOFC/SOEC). The absolute current $|I|$ is progressively increased to its first target value with a rate $\Delta I/\Delta t$ to be defined for each application, maintained to this value for a certain operating time $t_{op,1}$ and varied with the same rate to a second target value for another operating time $t_{op,2}$. A load cycle can comprise several plateaus at different current values and is then repeated m times.

Table 1: Static test input parameters during TM13.

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
Temperature of the oven	T_{oven}	°C	K
Temperature of the pre-heaters for preheating the negative electrode gas stream	$T_{PH,neg}$	°C	K
Temperature of the pre-heaters for preheating the positive electrode gas stream	$T_{PH,pos}$	°C	K
Rate of current change	$\Delta I/\Delta t$	$A\ s^{-1}$	$A\ s^{-1}$
Operating time of the plateau d	$t_{op,d}$	s, min, h	s
Number of plateaus per cycle	d	-	-
Number of cycles	m	-	-

Table 2: Variable test input parameters during TM13.

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
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 $l_n\ min^{-1}$, $l_s\ min^{-1}$	$m^3\ s^{-1}$
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	$x_{i,pos,in}$	-	-
Electrical current through the cell/stack	I	A	A

4 Test Output Parameters (TOPs)

Table 3 below shows the list of the test output parameters that are recorded during the overall duration of TM13, mainly the cell/stack assembly unit response e.g. V_{cell}, V_{stack} and $V_{RU,i}$ SOC voltages. In association with the current I and for long-term endurance tests, those TOPs allow the calculation of the SOC cell/stack performance degradation rates on voltage for each current plateau and as described in the master document TM00 (section 10).

T_{cell}, T_{stack}, T_{TP}, T_{BP}, $T_{neg,in}$, $T_{pos,in}$, $T_{neg,out}$ 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 in addition to OCV value). $f_{neg,out}$, $f_{pos,out}$, $f_{i,neg,out}$ 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 3: Test output parameters during TM13.

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 the repeating unit (RU) *i* in the stack	$V_{RU,i}$	V	V
Temperature of the cell	T_{cell}	°C	K
Temperature of the stack	T_{stack}	°C	K
Temperature of the top plate	T_{TP}	°C	K
Temperature of the bottom plate	T_{BP}	°C	K
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
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}
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}
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

The following *Table 4* gives the derived quantities useful for this TM. For each current plateau, they are all calculated from TIPs and TOPs with the equations presented in TM00 - section 10.

Table 4: Calculated derived quantities during TM13.

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
Electrical current density through the cell/stack	j	A cm^{-2}	A m^{-2}
Gas utilization at the negative electrode	$U_{gas,neg}$	-	-
Gas utilization at the positive electrode	$U_{gas,pos}$	-	-
Average RU voltage of all RUs in the stack	$V_{RU,av}$	V	V
Average temperature of the stack	T_{av}	°C	K
Degradation rate of cell	$\Delta V_{cell}/\Delta t$	V s^{-1}	V s^{-1}

Degradation rate of stack	$\Delta V_{stack}/\Delta t$	V s^{-1}	V s^{-1}
Degradation rate of repeating unit (RU) *i* voltage	$\Delta V_{RU,i}/\Delta t$	V s^{-1}	V s^{-1}

For long-term endurance test, the degradation rate is reported as the change in voltage (ΔV_{cell}, ΔV_{stack} and $\Delta V_{RU,i}$) over a given time which has to be clearly mentioned (see TM00 – section 10). Nevertheless, the first hundred hours are generally disregarded because the cell/stack assembly unit follows a kind of transient operation and the degradation rate can be very high in this period. The degradation rate is expressed in mV per kh (µV per h), as defined in section 10 of the master document TM00 and reference [2]. It can additionally be expressed in % of the initial voltage value. Moreover, it is either related to the operating time or the number of cycles.

The absolute degradation ΔX of a quantity X within the time from t_0 to t_1 is calculated as the difference between the final value $X(t_1)$ and the initial value $X(t_0)$:

$$\Delta X = X(t_1) - X(t_0)$$

The relative degradation ΔX_{rel} is calculated by dividing ΔX by the initial value $X(t_0)$:

$$\Delta X_{rel} = \frac{X(t_1) - X(t_0)}{X(t_0)} \cdot 100\%$$

The degradation rate (rate of change) of quantity X during the time interval $(t_1\text{-}t_0)$ is then calculated by:

$$\frac{\Delta X}{\Delta t} = \frac{\Delta X}{t_1 - t_0}$$ with the unit [unit of *X*/time unit]

$$\frac{\Delta X_{rel}}{\Delta t} = \frac{\Delta X_{rel}}{t_1 - t_0}$$ with the unit [%/time unit]

Degradation rates are typically expressed by the absolute or relative change per 1000 hours. It is thus advisable to normalize the results to 1000 h time interval. This can simply done by converting the unit of time interval to kh.

For load cycling experiments, degradation rate values can also be related to the number of cycles *m*:

$$\Delta X_m = \frac{X(t_1) - X(t_0)}{m}$$ and $$\Delta X_{m,rel} = \frac{X(t_1) - X(t_0)}{X(t_0) \cdot m} \cdot 100\%$$

6 Test Procedure

This specific TM is dedicated to the study of the capability of cell/stack assembly unit to modulate its power according to application-specific load profiles.

6.1 Critical Parameters and Parameter Controls

In this specific TM for operation under varying current and in addition to the stability of the operating conditions on each current plateau (temperature, pressure, gas flows and current density), a particular attention has to be paid to the accuracy of the current and its rate of change in order to follow the load patterns as close as possible. Also voltage measurement as a function of time has to be sufficiently accurate and voltage signal sufficiently clean to allow degradation rate determination.

Furthermore, it is important to avoid pollution from inlet gases and the test bench itself, since it has a strong influence on the degradation.

6.2 *Operation under varying current*

Realistic testing load profiles have to be defined for each application selected (stationary and distributed power generation (SOFC-µCHP), mobile SOFC-APU application, SOEC for H_2 production (power-to-gas) and combined SOFC/SOEC for electricity storage (power-to-gas-to-power) for instance). In the following subsections, typical load profiles from literature references for these applications are shown. These profiles can be used as examples in order to develop a simplified and practical test profile (as shown in *Figure 3*).

At a relevant temperature, inlet gas compositions and flow rates for each application, realistic loads I, plateau durations $t_{op,d}$ and speed rates for switching from one current to the other $\Delta I/\Delta t$ have to be defined based on literature and manufacturers' recommendations. Also the number of plateaus d and cycles m to be tested has to be fixed. Number of 200 cycles [2] and duration of 5 days [4] which can lead to about 500 cycles depending on the load pattern are mentioned in literature. Fuel/air utilization and steam conversion $U_{gas,neg}/U_{gas,pos}$ are mentioned for each tested load.

1) Check that the stability of the TIPs (T_{oven}, $T_{PH,neg}$, $T_{PH,pos}$, $p_{neg,out}$, $p_{pos,out}$, $f_{neg,in}$, $f_{pos,in}$, $f_{i,neg,in}$ *and* $f_{i,pos,in}$) is acceptable;

2) Increase $|I|$ from zero to the first targeted value progressively in accordance with the chosen rate of current application $\Delta I/\Delta t$;

3) Let operation last for $t_{op,1}$ then vary $|I|$ from the first to the second target current value with the chosen rate of current change $\Delta I/\Delta t$ for $t_{op,2}$ operation and so on. When the first cycle is finished, repeat it m times. When cycles last sufficiently long, EIS measurements can usefully be performed and are recommended between each full cycle. It is also recommended to measure and record properly all TIPs and TOPs mentioned in sections 3 and 4. In particular, the evolution of the V_{cell}, V_{stack} and $V_{RU,i}$ voltages and of the T_{cell} or T_{stack} is carefully recorded as a function of time during the overall duration of the TM13. If a leak is suspected at any moment during the test (unexpected temperature evolution, signal instabilities), stack operation can be interrupted by decreasing the current absolute value to zero and OCV value checked. Then, in any operational mode (SOFC, SOEC), an automatic cut-off voltage threshold may also be set, bringing immediately to step 4) even if the foreseen number of cycles m isn't achieved yet. Alternatively, a value of voltage degradation rate (decrease in SOFC mode and increase in SOEC mode) can be defined to stop the test module automatically even if the foreseen number of cycles m isn't achieved yet. An automatic cut-off criteria may also be chosen on T_{cell} or T_{stack};

4) If different from zero at the last plateau, decrease $|I|$ to zero progressively in accordance with the chosen rate of current application $\Delta I/\Delta t$.

Figure 1 below presents the general TIPs' evolution all along this TM for one SOEC or SOFC operating mode (negative or positive current) and combined SOEC/SOFC operation.

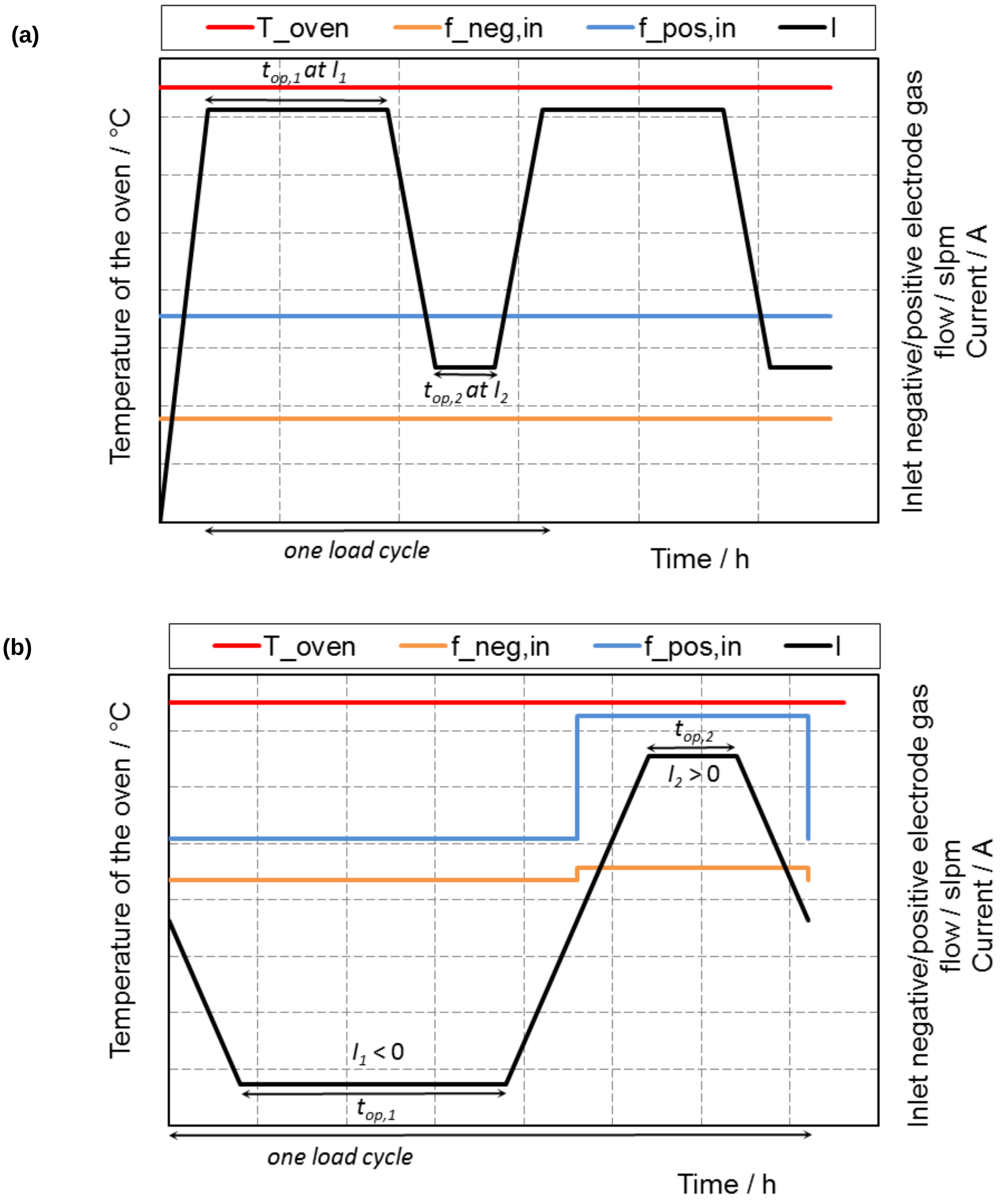


Figure 1: General evolution of TIPs during TM13 for (a) one SOEC or SOFC operating mode and (b) combined SOEC/SOFC operation.

On each current plateau, initial voltages and gas utilizations at the negative and positive electrodes (fuel utilization (SOFC) or steam conversion rate (SOEC) at the negative electrode and air / oxygen utilization (SOFC) at the positive electrode) are defined for each application.

6.2.1 For stationary and distributed power generation (SOFC-µCHP)

Traditionally the control strategy of µCHP units has focused on following either the electricity or the heat demand of the end-user. The irruption of smart and interconnected embedded control systems (IoT) allows operating the power units in an optimum (economic) point autonomously and this does not need to follow necessarily any of the loads.

Typical load and heat profiles are simulated in reference [2]. Simulation of SOFC operation for an electricity demand controlled unit with no export of electricity is shown in *Figure 2*. The green curves show the electricity demand and the red area is the electricity production in the SOFC unit.

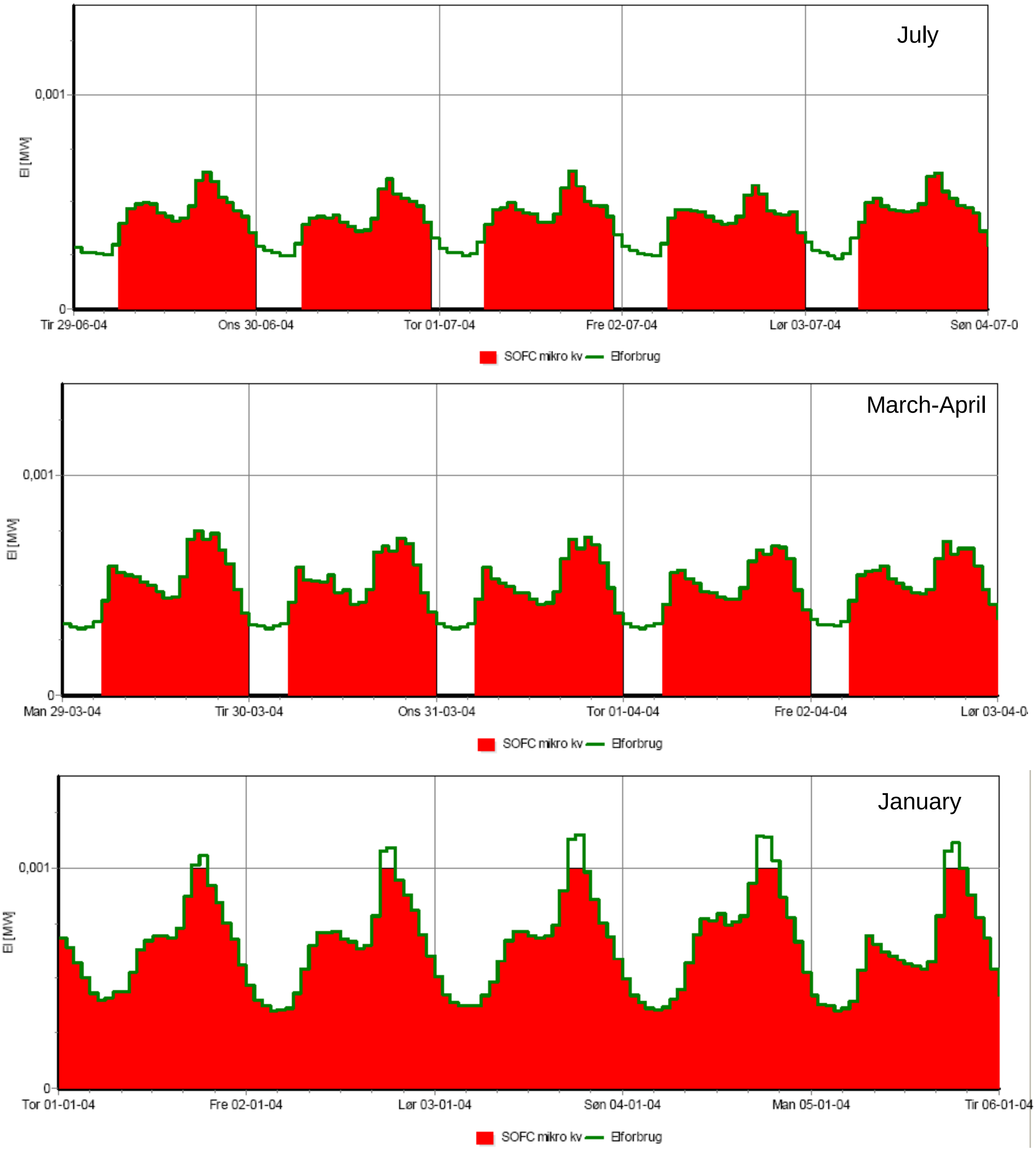


Figure 2: Example of load and heat profiles of a SOFC-µCHP (electricity demand controlled unit) [2]

In reference [2], the following simplified load pattern is proposed for SOFC tests simulating a micro-cogenerator in operation (see *Figure 3*).

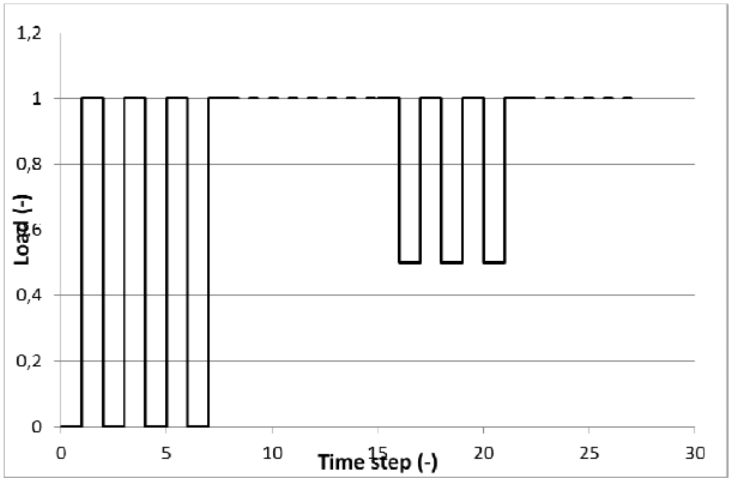
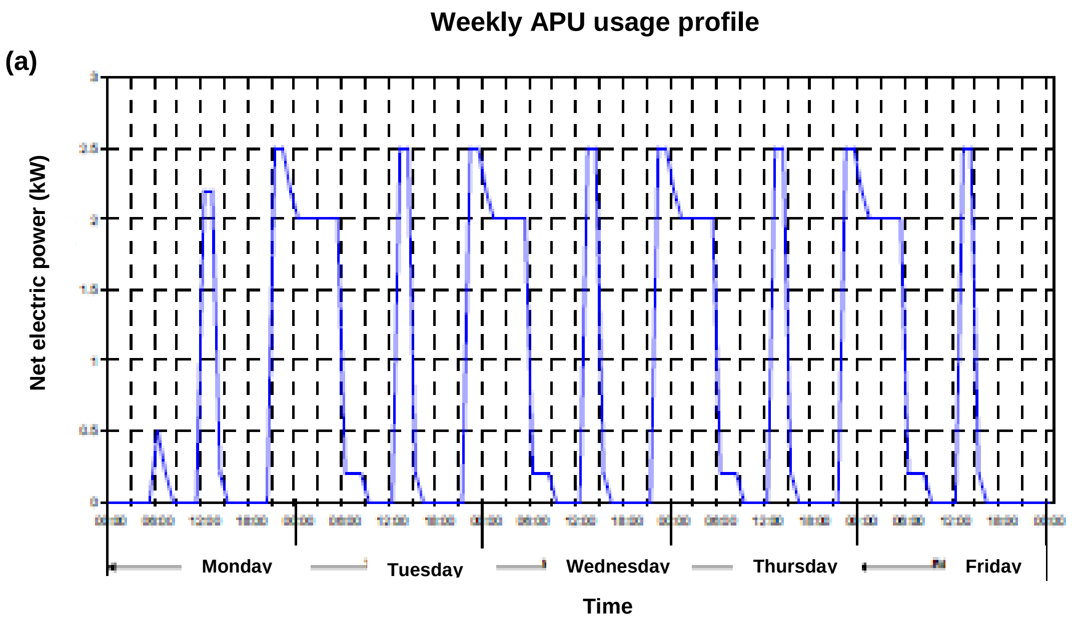


Figure 3: Simplified load profile simulating the operation of a SOFC-µCHP [2]

6.2.2 For mobile SOFC-APU application

A typical usage profile of a truck APU system is supplied in *Figure 4*(a) below with a focus on 48 hours in *Figure 4*(b) and can be used to define relevant testing load patterns.



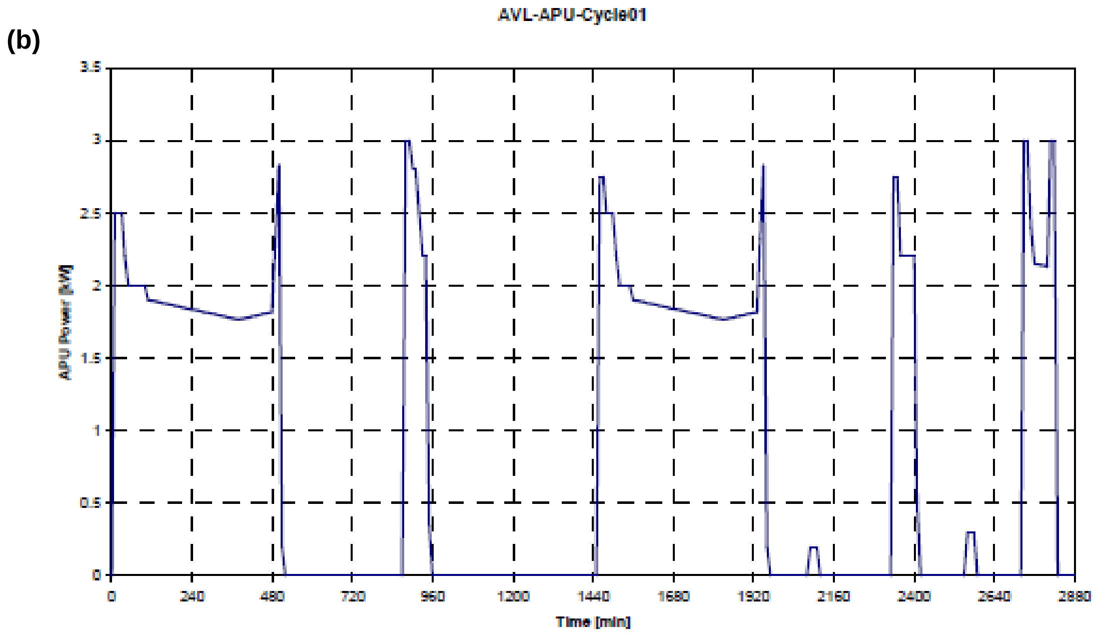


Figure 4: Load profile (a) and focus on 48 hours (b) of a SOFC truck APU [3]

6.2.3 Concerning SOEC for H_2 production (power-to-gas) and combined SOFC/SOEC for electricity storage (power-to-gas-to-power)

When coupling the SOC assembly unit with power generators relying on renewable energy sources, a realistic load profile can be defined based on reference [4] in SOEC mode. The following load profiles have been tested at single cell, SRU and short stack levels (see below) taking into account the renewable energy conversion technologies (converting solar or wind energy to electricity for instance). In order to study the influence of electric and/or thermal effects on degradation, testing of the cell/stack assembly unit is performed with different current densities corresponding to thermoneutral or exothermal operation (galvanostatic mode in *Figure 5* to *Figure 8*). Different rates of current change corresponding to ON/OFF operation of the electrolyser (highest value in *Figure 5* and *Figure 6*) or following load curves of renewable electricity production (lower value in *Figure 7* and *Figure 8*) are also interesting to be tested.

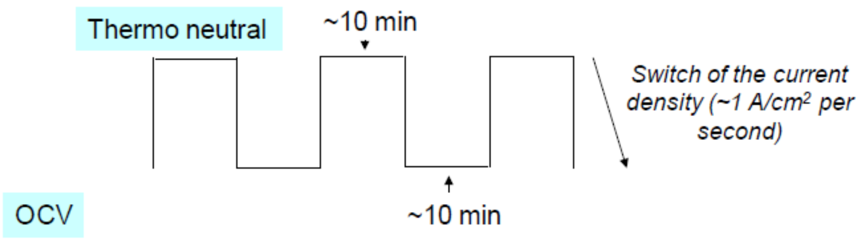


Figure 5: Load profile of a SOEC system with fast switch on/off at thermoneutral conditions [4]

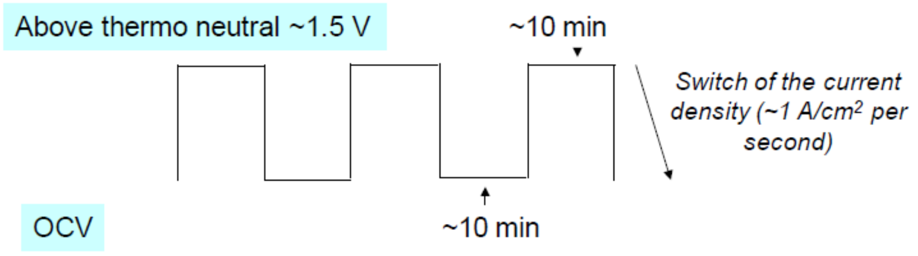


Figure 6: Load profile of a SOEC system with fast switch on/off at exothermal conditions [4]

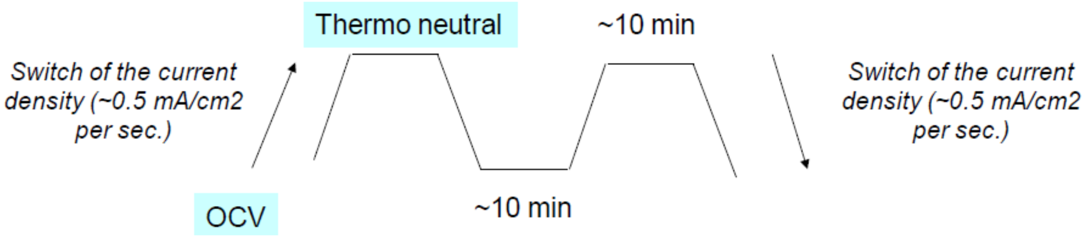


Figure 7: Load profile of a SOEC system with renewable energy source following and thermoneutral conditions [4]

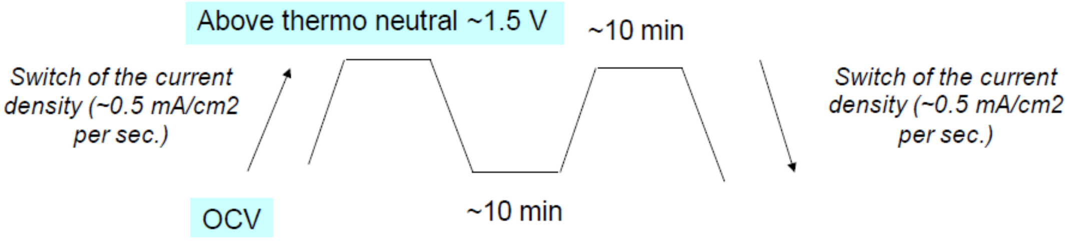


Figure 8: Load profile of a SOEC system with renewable energy source following and exothermal conditions [4]

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 presenting the evolution of V_{cell}, V_{stack} and $V_{RU,i}$ voltages as a function of time is highly recommended as well as a graph with the time-evolution of T_{cell} or T_{stack} (see *Figure 9* and *Figure 10* in SOEC and combined SOFC/SOEC modes for instance).

Also the calculated voltage degradation rates can be compared in a graph as shown in *Figure 11*.

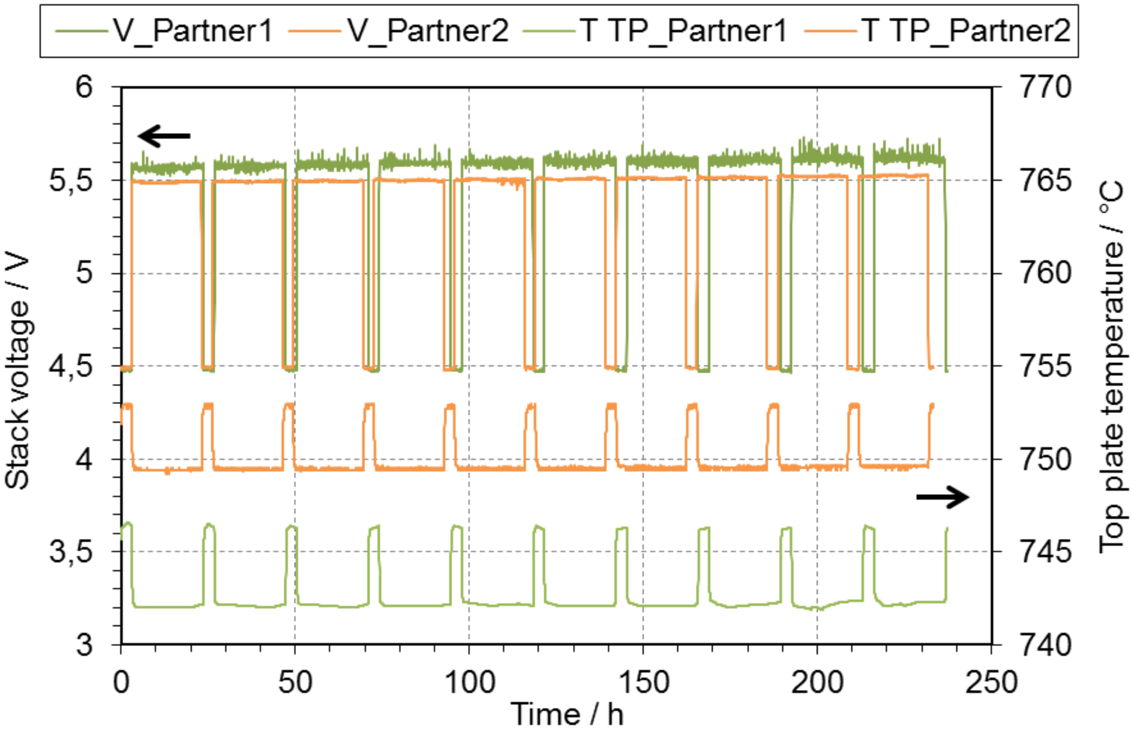


Figure 9: Examples of data representation in SOEC mode during TM13. Stack voltage and top plate temperature as a function of time.

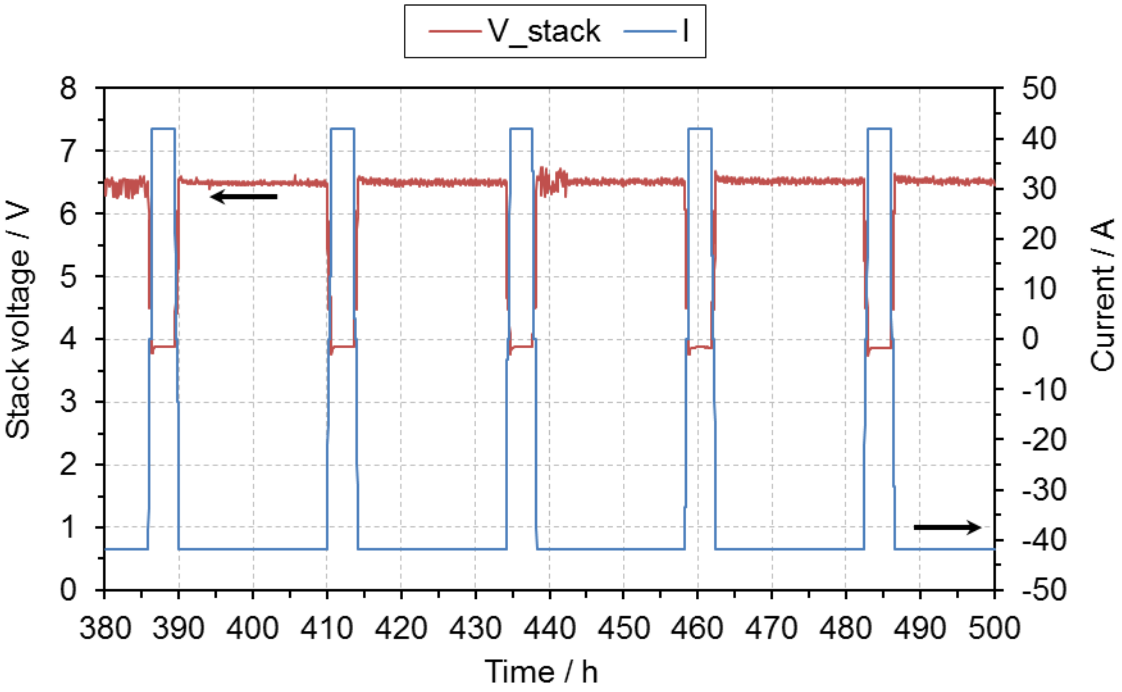


Figure 10: Example of data representation in combined SOFC/SOEC mode during TM13: Stack voltage and electrical current as a function of time.

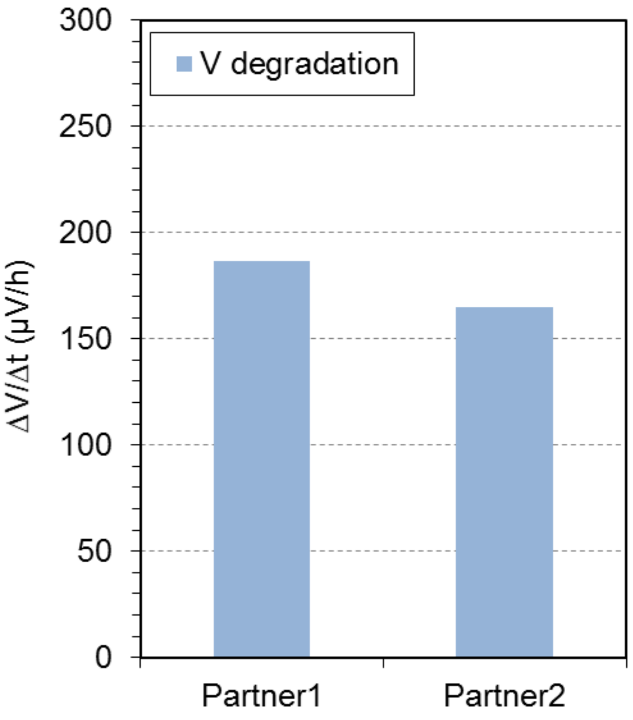


Figure 11: Example of data representation in SOEC mode during TM13: voltage degradation rates between two partners.

8 Differences to Existing Procedures

This TM objective is quite new. Few test programs in transient operating conditions can be found in literature [2-4] but existing procedures as reference [1] don't take into account dynamic operation in both modes. Based on those references, the present TM13 fully dedicated to operation under varying current presents in details the relevant TIPs, TOPs and derived quantities with their associated formularies, their evolutions, the test procedure to perform long-term operation in SOFC, SOEC and even combined SOFC/SOEC modes and the different ways to express degradation rates in order to achieve a more comprehensive representation of the cell/stack assembly unit durability.

9 Bibliography

[1] 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)

[2] M. Näslund, H. Iskov, Delrapport DGC1: Accelerated lifetime testing and standardization of SOFC systems, Danish Gas Technology Centre, Horsholm 2012

[3] EU - Project METSOFC - Grant agreement number: 211940 - Project final report - 2012, http://www.metsofc.eu/Publications/~/media/Metsofc/downloads/metsofc_final_report.ashx, latest access 12/12/2016

[4] ADEL deliverable D1.5 – Definition of ADEL testing protocol and revised test matrix, Q. Fu, A. Brisse, EIFER, 2013

Test Module 14

Thermal Cycling

(Version 2.5, 12 May 2017)

Objective and Scope

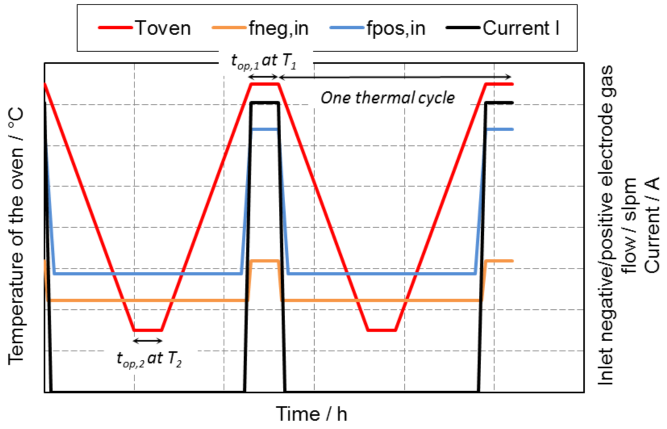
This test module deals with thermal cycling of solid oxide cell (SOC) either as a fuel cell (SOFC) or an electrolyser (SOEC), composed of several start-up/shut-down occurring for the overall SOC lifetime. It is a general characterization method that can be used in SOC R&D and for quality assurance.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Rate of oven temperature change ($\Delta T_{oven}/\Delta t$)	Temperature of the oven (T_{oven})
Dwell time at the plateau d ($t_{op,d}$)	Flow rates of inlet gases (f_{in})
Number of cycles and plateaus (m and d)	Composition of inlet gases ($x_{i,in}$)
Rate of current change ($\Delta I/\Delta t$)	Current (I)

Test Procedure

- Decrease temperature from the operating one under way to d plateau value at its specified rate of change.
- Wait for $t_{op,d}$ to elapse and continuously record all TIPs & TOPs at their specified sampling rates, e.g. 1 Hz.
- Continue with the next d+1 plateau value until the number m of cycles is exhausted.
- Operating periods and/or electrochemical measurements (j-V curve and EIS) can be usefully performed between each cycle.
- The test can be interrupted or terminated when operational abnormalities (such as unexpected temperature evolution, signal instabilities) are observed or certain predefined cut-off criteria are fulfilled (threshold values on voltage, temperature or degradation rate).



General evolution of TIPs during TM14 when temperature drops below 600°C with gas and current changes for instance.

Critical Parameters and Parameter Controls

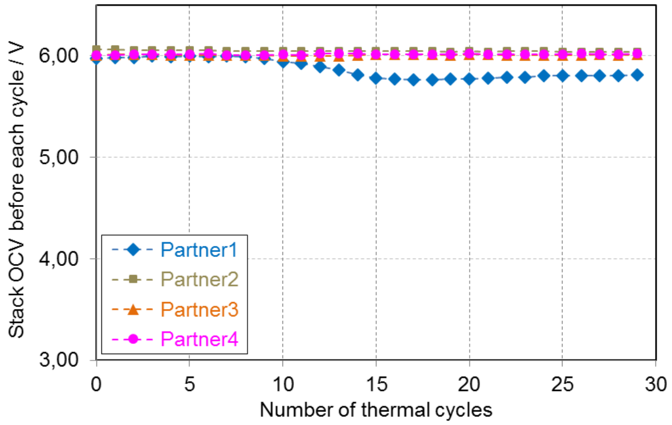
- The furnace thermal inertia very often limits the rate of the cooling down process which is then often lower than the heating up rate.
- When the lowest temperature is below 600°C, H_2 fraction at the negative electrode has to be kept below 4% to avoid explosive atmosphere.

Main Test Output Parameters (TOPs) and Derived Quantities

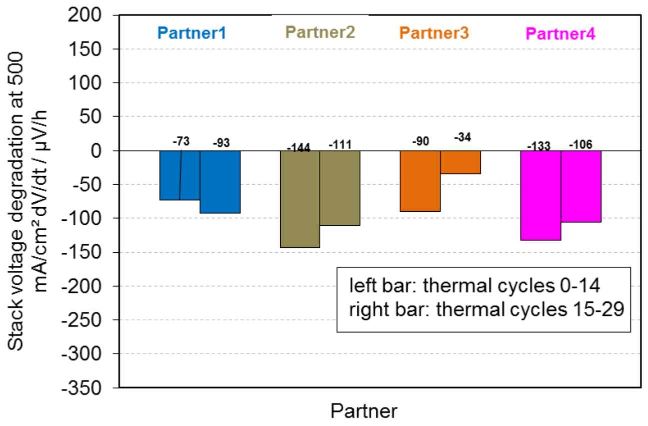
TOP	Derived Quantity
Voltage of cell/stack (V)	Current density (j)
Flow rates of outlet gases (f_{out})	Gas utilization (U_{gas})
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	Degradation rate of cell/stack voltage ($\Delta V/\Delta t$)
Composition of outlet gases ($x_{i,out}$)	Average temperature (T_{av})

Data Post Processing and Representation

Data representation examples under thermal cycling:



Evolution of stack OCV with cycle number.



Calculated stack voltage degradation rates at 0.5 A cm² during thermal cycling

SOCTESQA:
Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Project website: www.soctesqa.eu

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU GRANT AGREEMENT N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
***Start date**: 01.05.2014 – **Duration**: 36 months*
***Project Coordinator**: M. Lang – DLR*

Test Module 14: Thermal Cycling

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Version: 2.5
Last revision: 12.05.2017

Content

Abbreviations

APU	Auxiliary power unit
ASR	Area specific resistance
CHP	Combined heat and power
nlpm	Normal liter 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 liter per minute
TIP	Test input parameter
TM	Test module
TOP	Test output parameter

TM 14 – Thermal Cycling

1 Objective and Scope

This document presents the test module 14 (TM14) which deals with thermal cycling of solid oxide cell/stack assembly unit (SOC). Indeed, SOC assembly units either as fuel cell (SOFC) or as electrolyser (SOEC) are expected to bear thermal cycling during their overall lifetime depending on the application. These thermal cycles are usually used in test programs to represent several start-up/shut-down of the relevant SOC system. However, none of the previous projects (FCTESQA, FCTESTNET, Stacktest) nor the existing literature on test procedures for fuel/electrolysis cells [1] addresses the thermal cycling issue. Therefore, the aim of this test module is to establish a widely accepted method to evaluate stability of SOC when thermally cycled. A calculation method of the SOC degradation rate is also recommended from the continuous SOC voltages recording as a function of time. The particular interest of this method consists in achieving the voltage evolution/dependency over time and so to know if SOC performance evolves steadily or not. Other TMs, such as TM03 “Current-voltage characteristics” and TM04 “Electrochemical impedance spectroscopy”, also allow to calculate SOC degradation rates of cell/stack resistances (ohmic and polarization). It is necessary to provide different representations of degradation in order to give a comprehensive representation of the cell/stack assembly unit durability. This test module addresses SOC cell/stack assembly units, testing systems, instruments and measuring methods and test methods. Moreover, it is also applicable to the four different SOC applications selected in the SOCTESQA project which will influence the thermal patterns to be tested:

- Stationary and distributed power generation (SOFC-µCHP)
- Mobile (SOFC-APU)
- H_2 production in power-to-gas (SOEC)
- Electricity storage in power-to-gas-to-power (SOEC/SOFC)

Definition of relevant thermal profiles related to these applications are supported by recommendations of the SOCTESQA Industrial Advisory Board members. All the quantities used in TM14 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 detailed described in chapter 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 practical guidance 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)

There are two types of test input parameters: variable ones which vary during the TM duration according to the chosen application and static ones which don’t vary during the overall duration of the TM. This

TM is carried out from the operating temperature under way, controlled through T_{oven} setpoint, to lower ones at a constant rate of temperature change $\Delta T_{oven}/\Delta t$ during a number of cycles m. $t_{op,d}$ is the dwell time at each thermal plateau d of one cycle. As some operating periods can be integrated from time to time between thermal cycles (see Section 6.3), I and $\Delta I/\Delta t$ appear as TIPs in this TM.

Table 1 and *Table 2* below list all the test input parameters (operating conditions) which have to be controlled in this TM.

Table 1: Static test input parameters during TM14.

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
Rate of oven temperature change	$\Delta T_{oven}/\Delta t$	°C s^{-1}	K s^{-1}
Number of plateaus per cycle	d	-	-
Number of cycles	m	-	-
Dwell time of the plateau *d*	$t_{op,d}$	s, min, h	s
Rate of current change	$\Delta I/\Delta t$	A s^{-1}	A s^{-1}

Table 2: Variable test input parameters during TM14.

Description of quantity	**Symbol**	**Unit often used**	**SI unit**
Temperature of the oven	T_{oven}	°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}
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	$x_{i,pos,in}$	-	-
Electrical current through the cell/stack	I	A	A

4 Test Output Parameters (TOPs)

Table 3 below shows the list of the test output parameters which are recorded during the overall duration of this TM. These are mainly parameters related to SOC tightness like temperatures (T_{cell}, T_{stack}, T_{TP},

T_{BP}, $T_{neg,in}$, $T_{pos,in}$, $T_{neg,out}$ and $T_{pos,out}$, $f_{neg,out}$, $f_{pos,out}$, $f_{i,neg,out}$ and $f_{i,pos,out}$, $x_{i,neg,out}$ and $x_{i,pos,out}$, $p_{i,neg,out}$ and $p_{i,pos,out}$) and OCVs but also performance parameters like voltages at operating current I (V_{cell}, V_{stack}, $V_{RU,i}$). Those parameters will allow the evaluation of the SOC cell/stack thermal stability and the calculation of its leak and degradation rates.

Table 3: Test output parameters during TM14.

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 the repeating unit (RU) *i* in the stack	$V_{RU,i}$	V	V
Temperature of the cell	T_{cell}	°C	K
Temperature of the stack	T_{stack}	°C	K
Temperature of the top plate	T_{TP}	°C	K
Temperature of the bottom plate	T_{BP}	°C	K
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
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}
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}
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

The following *Table 4* gives the derived quantities useful for this TM, depending on the thermal cycling method mentioned in Section 6.3. The derived quantities are all calculated from TIPs and TOPs with the equations presented in TM00 - Section 10.

For the continuous thermal cycling above 600°C (see *Figure 1*), only $V_{RU,av}$, $\Delta V_{cell}/\Delta t$, $\Delta V_{stack}/\Delta t$ *and* $\Delta V_{RU,i}/\Delta t$ are followed at zero current. This does not allow determining performance degradation but only the degradation of the OCVs. This gives an indication of the change of the gas tightness of the cell/stack assembly unit during thermal cycling. Sometimes, the degradation values are related to the number of cycles *m*.

For the thermal cycling with operation at constant current periods (see *Figure 2*), also the change of the voltage values $\Delta V_{cell}/\Delta t$, $\Delta V_{stack}/\Delta t$ *and* $\Delta V_{RU,i}/\Delta t$ under electrical current is analyzed. This results in degradation values which are either related to the operating time or the number of cycles. For coupling of thermal cycles with electrochemical performances (jV-curve and/or EIS diagram) on each plateau at the operating temperature, $U_{gas,neg}$ and $U_{gas,pos}$, *ASR*, $P_{d,el}$, $\Delta ASR/\Delta t$ and $\Delta P_{d,el}/\Delta t$ might be useful values to express degradation behavior.

Table 4: Calculated derived quantities during TM14.

Description of quantity	Symbol	Unit often used	SI unit
Electrical current density through the cell/stack	j	A cm^{-2}	A m^{-2}
Gas utilization at the negative electrode	$U_{gas,neg}$	-	-
Gas utilization at the positive electrode	$U_{gas,pos}$	-	-
Average RU voltage of all RUs in the stack	$V_{RU,av}$	V	V
Area specific resistance	*ASR*	Ω cm^2	Ω 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}
Degradation rate of cell	$\Delta V_{cell}/\Delta t$	V s^{-1}	V s^{-1}
Degradation rate of stack	$\Delta V_{stack}/\Delta t$	V s^{-1}	V s^{-1}
Degradation rate of repeating unit (RU) *i* voltage	$\Delta V_{RU,i}/\Delta t$	V s^{-1}	V s^{-1}
Degradation rate of cell/stack/RUs ASR	$\Delta ASR/\Delta t$	Ω cm^2 s^{-1}	Ω m^2 s^{-1}
Degradation rate of cell/stack/RUs electrical power density	$\Delta P_{d,el}/\Delta t$	W s^{-1} cm^{-2}	J s^{-2} m^{-2}

The absolute degradation ΔX of a quantity X within the time from t_0 to t_1 is calculated as the difference between the final value $X(t_1)$ and the initial value $X(t_0)$:

$$\Delta X = X(t_1) - X(t_0) \tag{1}$$

The relative degradation ΔX_{rel} is calculated by dividing ΔX by the initial value $X(t_0)$:

$$\Delta X_{rel} = \frac{X(t_1) - X(t_0)}{X(t_0)} \cdot 100\% \tag{2}$$

The degradation rate (rate of change) of quantity X during the time interval *(t_1-t_0)* is then calculated by:

$$\frac{\Delta X}{\Delta t} = \frac{\Delta X}{t_1 - t_0} \quad \text{with the unit [unit of } X\text{/time unit]} \tag{3}$$

$$\frac{\Delta X_{rel}}{\Delta t} = \frac{\Delta X_{rel}}{t_1 - t_0} \quad \text{with the unit [\%/time unit]} \tag{4}$$

Degradation rates are typically expressed by the absolute or relative change per 1000 hours. It is thus advisable to normalize the results to 1000 h time interval. This can simply done by converting the unit of time interval to kh.

For thermal cycling experiments, degradation values can also be related to the number of cycles *m*:

$$\Delta X_m = \frac{X(t_1) - X(t_0)}{m} \quad (5) \qquad \text{and} \qquad \Delta X_{m,rel} = \frac{X(t_1) - X(t_0)}{X(t_0) \cdot m} \cdot 100\% \quad (6)$$

6 Test Procedure

6.1 Critical Parameters and Parameter Controls

In this specific TM about thermal cycling, a particular attention has to be paid to the accuracy of the temperature and its rate of change in order to follow the thermal profiles of the system as close as possible. It is worth noticing here that in practice, the cooling down process is very often slower than the heating up one due to furnace thermal inertia which limits the cooling down speed. Also it is recommended to measure continuously relevant parameters as the ratio $f_{neg,out}$ / $f_{neg,in}$ and $f_{pos,out}$ / $f_{pos,in}$ or T_{cell}, T_{stack}, T_{av} or simply V_{cell}, V_{stack}, $V_{RU,i}$ at open circuit as long as temperature is high, in order to follow SOC cell/stack tightness through thermal cycling. To check OCV values of all repeating units before the thermal cycling is recommended as a less gas tight repeating unit in the stack may have a significant influence on the OCV of the other repeating units during thermal cycling. Moreover, it is relevant to check if the compression force F_{compr} is kept constant all along thermal cycling to maintain a good stack behavior (electrical contact and tightness).

For safety reason, when the minimum temperature is below about 600°C, H_2 fraction $x_{H2,neg,in}$ on the negative electrode side has to be kept below 4% to avoid explosive atmosphere.

6.2 Preconditioning of the Stack

Ideally, preconditioning parameters are supplied by the manufacturer. Nevertheless, a common procedure is described in the master document TM00.

6.3 Thermal cycling

Realistic thermal profiles have to be defined for each application. **For stationary and distributed power generation (SOFC-µCHP)**, a profile between the operating temperature and room temperature (< 50°C) with 25 thermal cycles is proposed in reference [2].

For mobile SOFC-APU application, a typical thermal profile of a truck APU system is used in reference [3] to define a relevant testing thermal pattern. Deep and medium thermal cycles are defined with the minimum temperature lower or higher than 100°C respectively. The deep ones consist of full cool down with a one week occurrence. The medium ones correspond to phases when no current is requested from the user so with a higher occurrence. Additionally, in reference [4] and [5] start-up and electrical power profiles are reported.

Figure 1 and *Figure 2* below present the general TIPs' evolution all along this TM. Concerning the operating conditions, there are three options during thermal cycling:

1) During thermal cycling the inlet positive and negative electrode gas flows ($f_{neg,in}$ and $f_{pos,in}$) and the inlet negative and positive electrode gas compositions ($x_{i,neg,in}$ and $x_{i,pos,in}$) are kept constant (see *Figure 1*). This operating mode is only possible if the cell/stack temperature during thermal cycling does not drop below 600°C. The electrical current is usually kept at zero. Without electrical current load, it is not possible to determine any electrochemical performance data but only the OCV of the cell/stack assembly unit. It is therefore recommended to regularly apply other test modules, e.g. TM03 (jV-curve) and/or TM04 (Electrochemical impedance spectroscopy), after several thermal cycles.

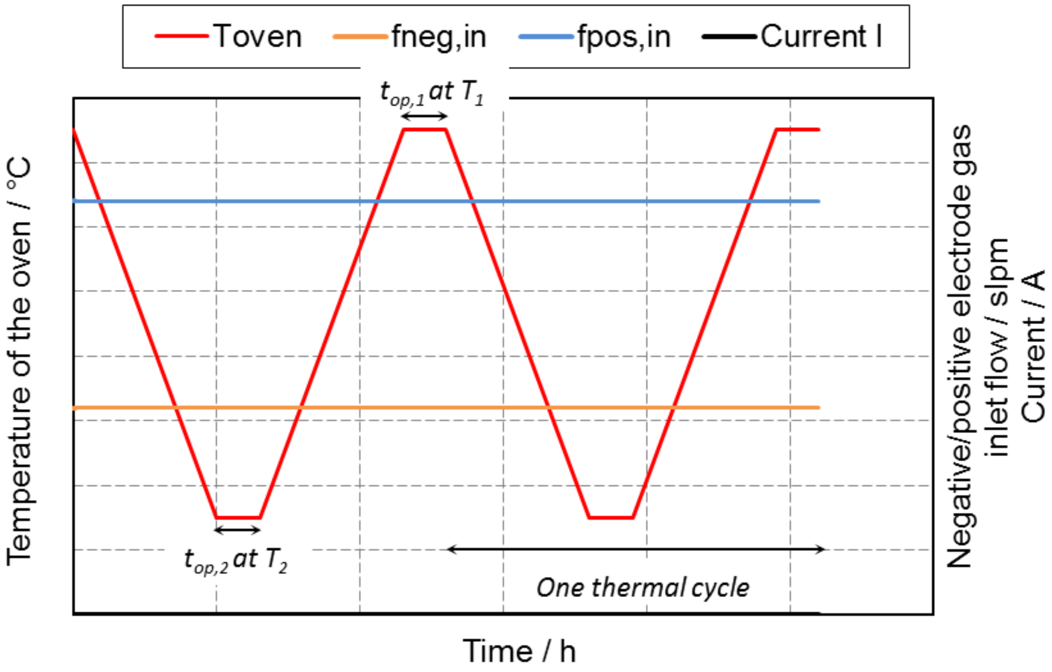

Figure 1: General evolution of TIPs during TM14: Continuous thermal cycling above 600°C (in this case with electrical current at zero)

2) During thermal cycling the inlet positive and negative electrode gas flows ($f_{neg,in}$ and $f_{pos,in}$) and the inlet negative and positive electrode gas compositions ($x_{i,neg,in}$ and $x_{i,pos,in}$) are changed (see *Figure 2*). This operating mode is necessary if the cell/stack temperature during thermal cycling drops below 600°C. Because of safety reasons below this temperature the H_2-concentration in the fuel gas has to be decreased to less than 4%. Usually, at high temperature the electrical current is increased and then kept at a constant level for a system relevant period. Usually this period for mobile applications is in the range of several hours and much shorter compared to the long operating period for stationary applications (see TM12: Operation at constant current). This allows

determining the evolution of the OCV, the performance data and the corresponding degradation of the cell/stack assembly during thermal cycling with limited effort. However, it is recommended to regularly apply other test modules, e.g. TM03 (jV-curve) and/or TM04 (Electrochemical impedance spectroscopy), after several thermal cycles [6].

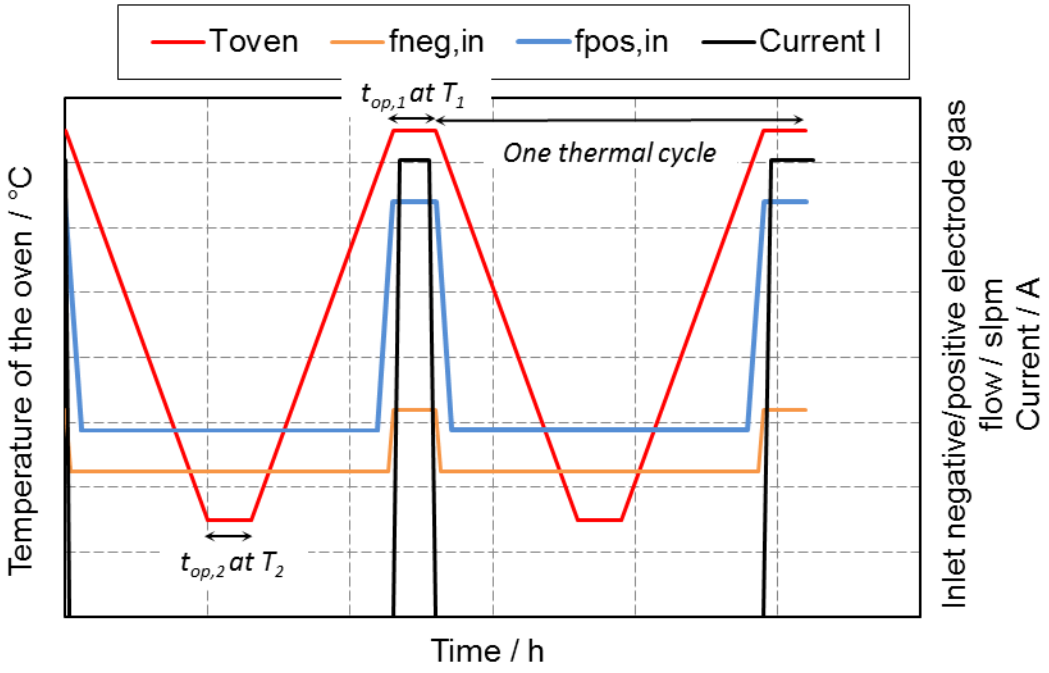

Figure 2: General evolution of TIPs during TM14: Thermal cycling below 600°C with gas and current changes (coupling with operation at constant current for instance).

3) When a precise following of the stack performance is required at the operating temperature from time to time along the TM, it is also possible to couple thermal cycling with electrochemical measurements. In this case the application of TM03 (jV-curve) and/or TM04 (Electrochemical impedance spectroscopy) might be useful. In this case, the inlet positive and negative electrode gas flows ($f_{neg,in}$ and $f_{pos,in}$), inlet negative and positive electrode gas compositions ($x_{i,neg,in}$ and $x_{i,pos,in}$), and current I are variable TIPs. This thermal cycling mode is very time consuming and delivers an entire set of performance and impedance data for each cycle. This method is therefore only recommended if significant degradation rates between the different thermal cycles are expected.

The test module starts at the operating temperature under way. Realistic plateau temperatures, plateau duration $t_{op,d}$ and speed rate for cooling-down and heating-up $\Delta T_{oven}/\Delta t$ have to be defined based on literature and manufacturers recommendations. Also the total duration or the number of plateaus per cycle d and thermal cycles m to be tested have to be fixed. Numbers of 25 [2] to 3000 cycles [3] are mentioned in literature.

Compressed testing (shorter duration but with same values of temperature and rate of temperature change as reality) will be preferred to accelerated ones (harsh conditions) mainly in order to keep same degradation mechanisms as in real operation.

The evolution of $f_{neg,out}$ / $f_{neg,in}$ and $f_{pos,out}$ / $f_{pos,in}$ or T_{cell}, T_{stack}, T_{av} or simply V_{cell}, V_{stack}, $V_{RU,i}$ at open circuit is recorded as a function of time during the overall duration of the TM. Anyway, a value of tightness degradation evaluated through these parameters can be defined to stop the test even if the foreseen duration or number of cycles is not achieved yet. Moreover if coupling with electrochemical performance is applied through jV-curve and EIS measurements or constant current operation from time to time all along the TM, the derived quantities mentioned in the previous section 5 can be usefully calculated as a function of time.

7 Data Post Processing and representation

Data post-processing consists in the analysis of the test output parameters and derived quantities evolution during the overall duration of the TM (as a function of time or cycle number). Usually, degradation rates are the most interesting results of thermal cycling experiments. Figure , 4, 5 and below are examples for representation of results for thermal cycling testing.

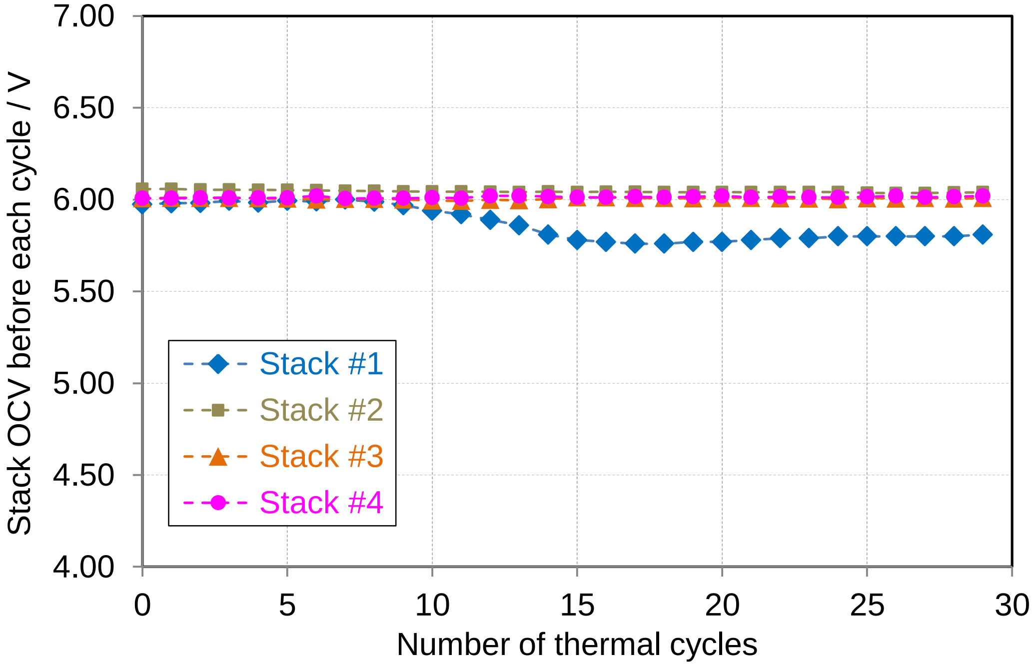

Figure 3: Stack OCVs at 750°C during 29 thermal cycles between 50°C and 750°C

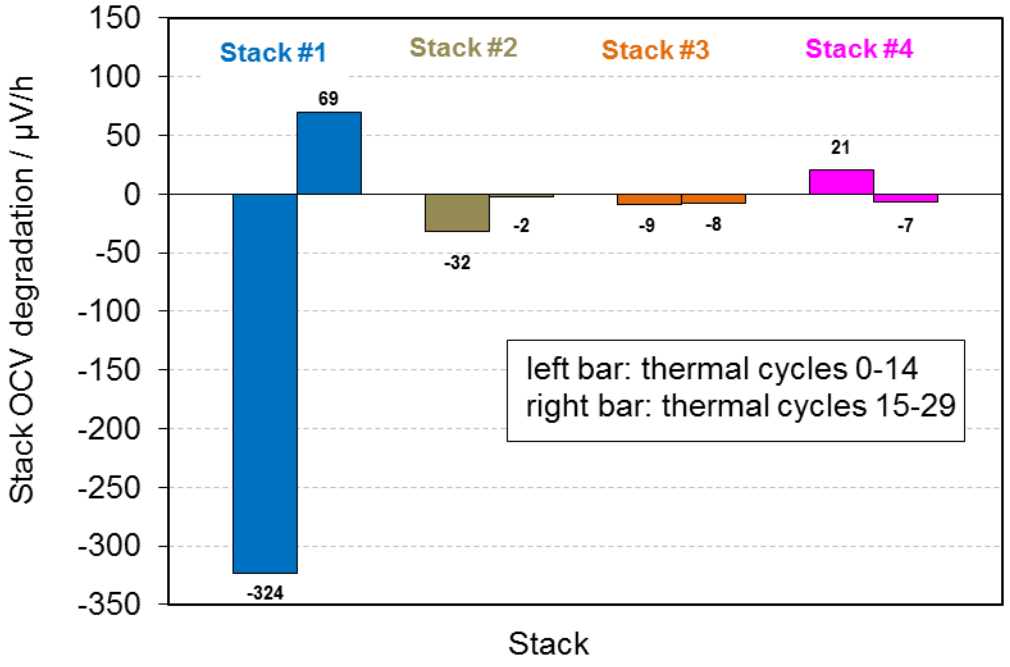

Figure 4: Calculated degradation of the stack OCVs during first 14 and second 15 thermal cycles between 50°C and 750°C

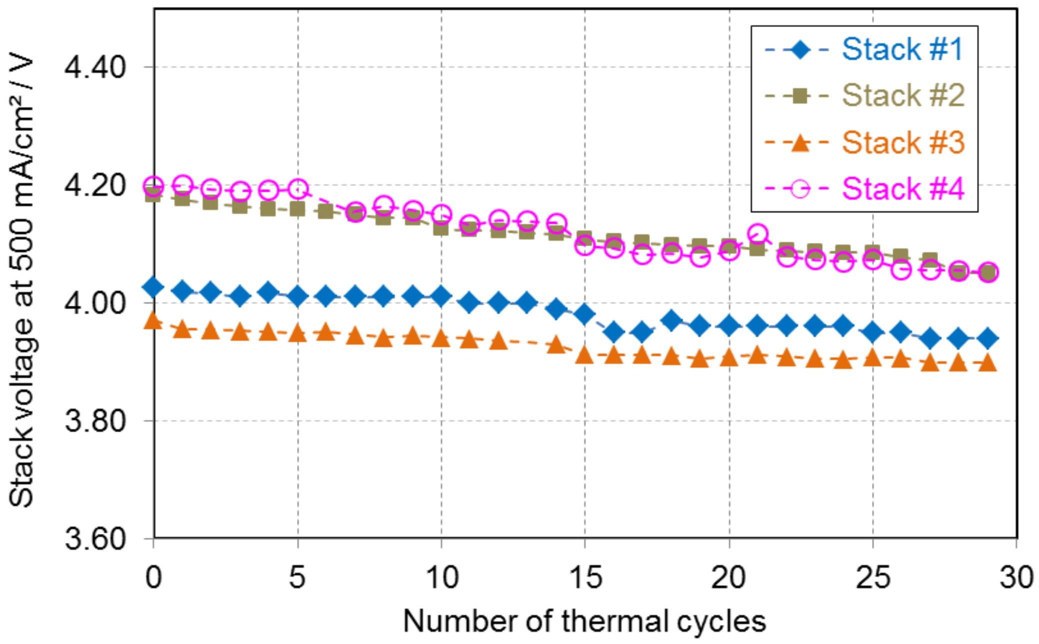

Figure 5: Stack voltage at 750°C and 500 mA cm^{-2} during 29 thermal cycles between 50°C and 750°C

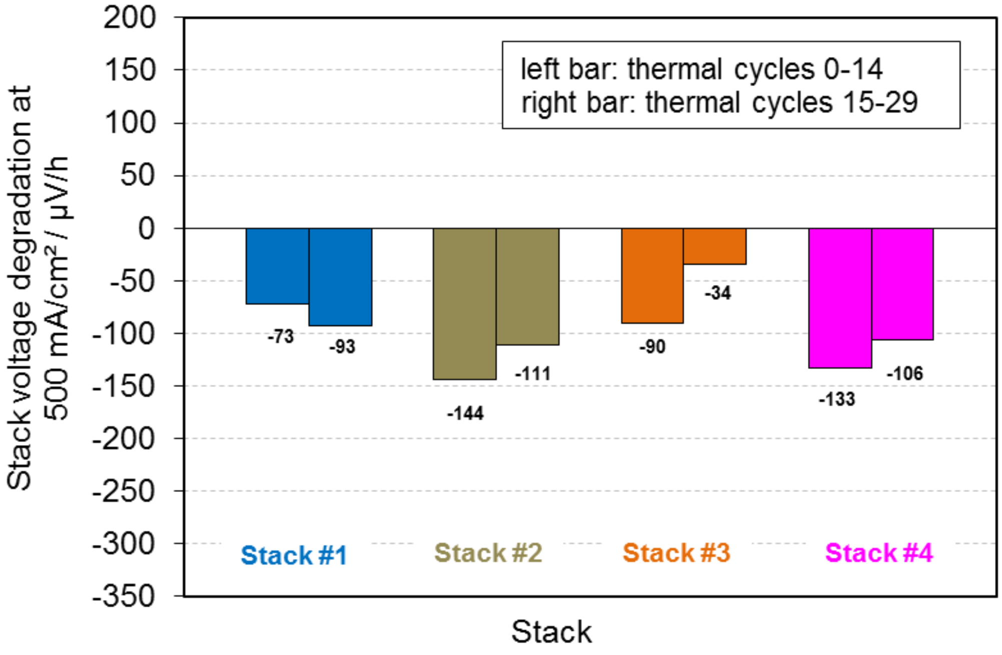

Figure 6: Calculated degradation of the stack voltage at 750°C and 500 mA cm^{-2} during first 14 and second 15 thermal cycles between 50°C and 750°C

8 Differences to Existing Procedures

This TM topic is quite common nowadays as shown by references found in literature [2-5]. Nevertheless, existing procedure as reference [1] remains quite generic. Based on those references, the present TM14 fully dedicated to thermal cycling presents in details the relevant TIPs, TOPs and derived quantities with their associated formularies, their evolutions, the different test procedure options to perform long-term thermal cycling in both SOFC and SOEC conditions and the different ways to express degradation rates in order to achieve a more comprehensive representation of the cell/stack assembly unit durability.

9 Bibliography

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Test Module 16

Shut-down

(Version 2.0, 10 May 2017)

Objective and Scope

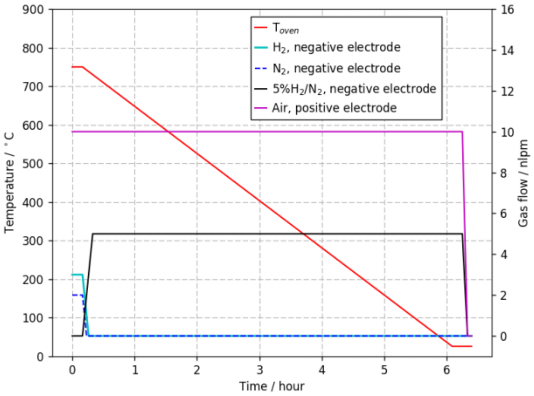
This test module deals with the shutdown procedures needed for termination of the SOC cell/stack operation and coming back to ambient temperature. The shutdown procedure normally should be given by the manufacturer. However, if there is no shutdown procedure available, a recommendation is given in this test module.

Main Test Input Parameters (TIPs)

Static TIP	Variable TIP
Rate of oven temperature change ($\Delta T_{oven}/\Delta t$)	Flow rates of inlet gases (f_{in})
Rate of the pre-heaters temperature change($\Delta T_{PH,in}/\Delta t$)	Temperature of the oven (T_{oven}) and pre-heaters ($T_{PH,in}$)
	Pressure of outlet gases (p_{out})
	Composition of inlet gases ($x_{i,\ in}$)

Test Procedure

- Decrease/increase the current I (voltage V) stepwise to zero current (OCV) in a galvanostatic (or potentiostatic) mode if applicable, and disconnect/switch off the electronic load and power supply.
- Change the reactant flow $f_{\mathrm{neg,in}}$ and $f_{\mathrm{pos,in}}$ to nominal values and $x_{i,neg,in}$ and $x_{i,pos,in}$ to nominal composition.
- Decrease the reactant pressures (both at the same time) $p_{\mathrm{neg,in}}$ and $p_{\mathrm{pos,in}}$ to ambient pressure, if applicable.
- Set negative electrode and positive electrode to protection gas (e.g. 5 % H_2 in N_2) and air at $f_{\mathrm{neg,in}}$ and $f_{\mathrm{pos,in}}$ respectively.
- Reduce cell/stack temperature T_{cell} / T_{stack} by adjusting pre-heaters temperature (if applicable) and T_{oven} with pre-defined temperature changing rates to ambient temperature.
- At ambient temperature, reduce gas-flows of negative and positive electrodes to zero.



Example of changing of temperature (T_{oven}) and gas flows of a 5-cell stack during shutdown.

Critical Parameters and Parameter Controls

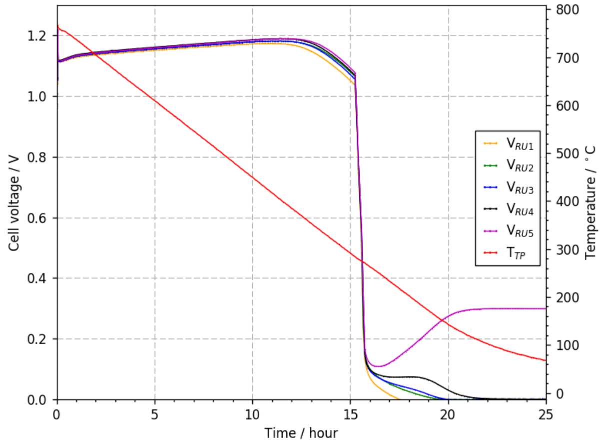
- During shutdown, the negative electrode gas is switched from H_2 rich gas to a diluted H_2 -mixture (protection gas, e.g. 5% H_2 in N_2).

Main Test Output Parameters (TOPs) and Derived Quantities

TOP	Derived Quantities
Voltage of cell/stack (V)	Average temperature of the stack (T_{av})
Temperature of gas streams at cell/stack inlet/outlet, temperature of cell/stack (T)	Maximum temperature difference during shutdown (ΔT_{max})

Data Post Processing and Representation

Representation examples of shutdown:



Stack temperature and RU voltages during shut-down of a SOC 5 cell stack test (Shutdown from SOFC conditions).

The research leading to these results has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 621245.

Solid Oxide Cell and Stack Testing, Safety and Quality Assurance

Collaborative Project - *FCH JU GRANT AGREEMENT N° 621245*
THEME [SP1-JTI-FCH.2013.5.4]
***Start date**: 01.05.2014 – **Duration**: 36 months*
***Project Coordinator**: M. Lang – DLR*

Test Module 16: Shut-down

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Version: 2.0
Last revision: 10.05.2017

Content

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 16 – Shut-down

1 Objective and Scope

The objective of this test module is to define the shut-down conditions needed for ending of the SOC cell/stack operation and coming back to the ambient temperature.The shut-down procedure normally should be given by the manufacturer. However, if no shut-down is available a recommendation is given in this test module (TM).

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)

There are two types of test input parameters: variable and static. The first type may vary during the duration of the TM while the second type does not vary during the overall duration of the TM. During shutdown, the stack/cell is cooling down by decreasing the temperature of the stack/cell test oven $\mathrm{T_{oven}}$ and/or pre-heaters $T_{PH,neg}$ *and* $T_{PH,pos}$ to ambient temperature. The temperature change rate of the SOC cell/stack test set-up normally is controlled by setting the heat/cooling rate $\Delta T_{oven}/\Delta t$ of *the oven*. $\Delta T_{oven}/\Delta t$ is also dependent on the test set-up, though generally 1 K $\mathrm{min^{-1}}$ for single cell testing [1, 3] is used.

The relevant static and variable test input parameters for the shut-down (TM 16) are given in *Table 1* and *Table 2*.

Table 1: Static test input parameters during TM 16.

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

Table 2: Variable test input parameters during TM 16.

Description of quantity	Symbol	Unit often used	SI Unit
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} (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	$x_{i,pos,in}$	-	-
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

4 Test Output Parameters (TOPs)

The main output parameters of this test are the cell/stack voltage and the voltages of the single repeating units (RUs) given in *Table 3*.

Table 3: Test output parameters for TM 16.

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 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	T_{TP}	°C	K
Temperature of the bottom plate of the stack	T_{BP}	°C	K
Temperature of the cell	T_{cell}	°C	K
Maximum temperature difference during shut-down	ΔT_{max}	°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 TM16.

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:

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

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

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

6 Test Procedure

If no shut-down procedure is provided by the stack manufacturer the following procedure is recommended to shut-down the SOC cell/stack.

6.1 Critical parameters and parameter controls

Figure 1 shows an example of an SOC stack shut-down procedure. During shut-down, the negative electrode gas is switched from H_2 rich gas mixture (in this example, 60% H_2 + 40% N_2) to protection gas (e.g. 5% H_2 in N_2). The air supplied to the positive electrode is kept at 10 nlpm. All the gases are cut off after the stack cooling down to ambient temperature.

6.2 Shut-down

1) Decrease the current I (voltage V) stepwise to zero current (OCV) in a galvanostatic (or potentiostatic) mode if applicable.
2) Disconnect/switch off the electronic load and power supply.
3) Change the reactant flow $f_{\mathrm{neg,\ in}}$ and $f_{\mathrm{pos,\ in}}$ to nominal values and $x_{i,neg,in}$ to nominal composition.
4) Decrease the reactant pressures (both at the same time) $p_{\mathrm{neg,\ in}}$ and $p_{\mathrm{pos,\ in}}$ to ambient pressure, if applicable.
5) Set negative electrode and positive electrode under H_2/N_2 (Ar) / air at $f_{\mathrm{neg,\ in}}$ and $f_{\mathrm{pos,\ in}}$ required by the setup respectively.
6) Reduce cell/stack temperature $T_{\mathrm{cell/stack}}$ by the predefined temperature change rate of $\Delta T_{oven}/\Delta t$. The gas inlet temperatures $T_{\mathrm{neg,\ in}}$ and $T_{\mathrm{pos,\ in}}$ are decreased (if possible) to ambient temperature by adjusting $T_{ph\ \mathrm{neg}}$ and $T_{ph\ \mathrm{pos}}$.
7) At ambient temperature reduce gas-flows of negative and positive electrodes to zero.

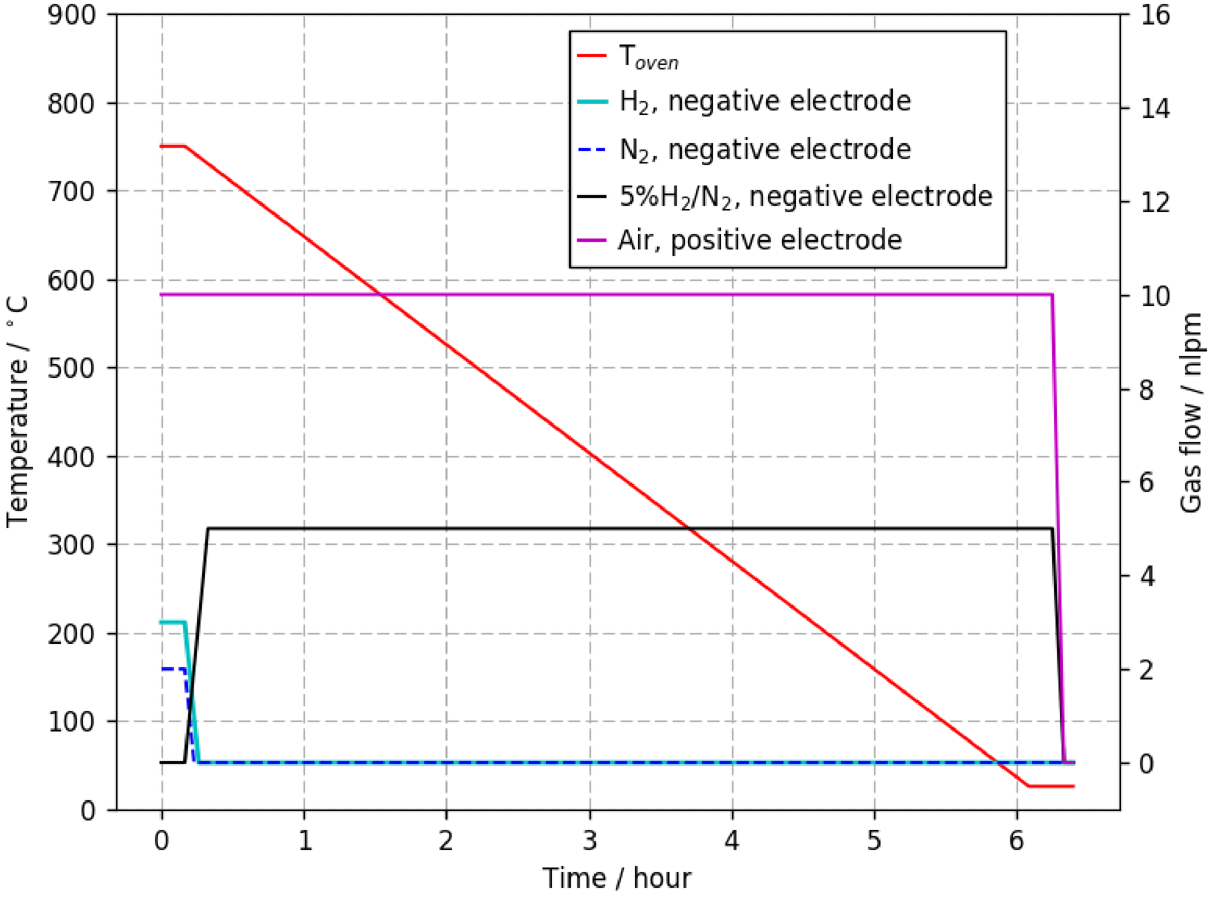

Figure 1: Schematic overview of a possible TM shut-down for a 5 cells SOC stack (from SOFC conditions).

7 Data Post Processing

Data representation of selected TOPs can be used as an overview of the performed TM. Figure 2 shows an example of RU voltages and temperature evolution during shut-down of a 5 cells SOC stack from SOFC testing condition (H_2 rich gas composition on the negative electrode).

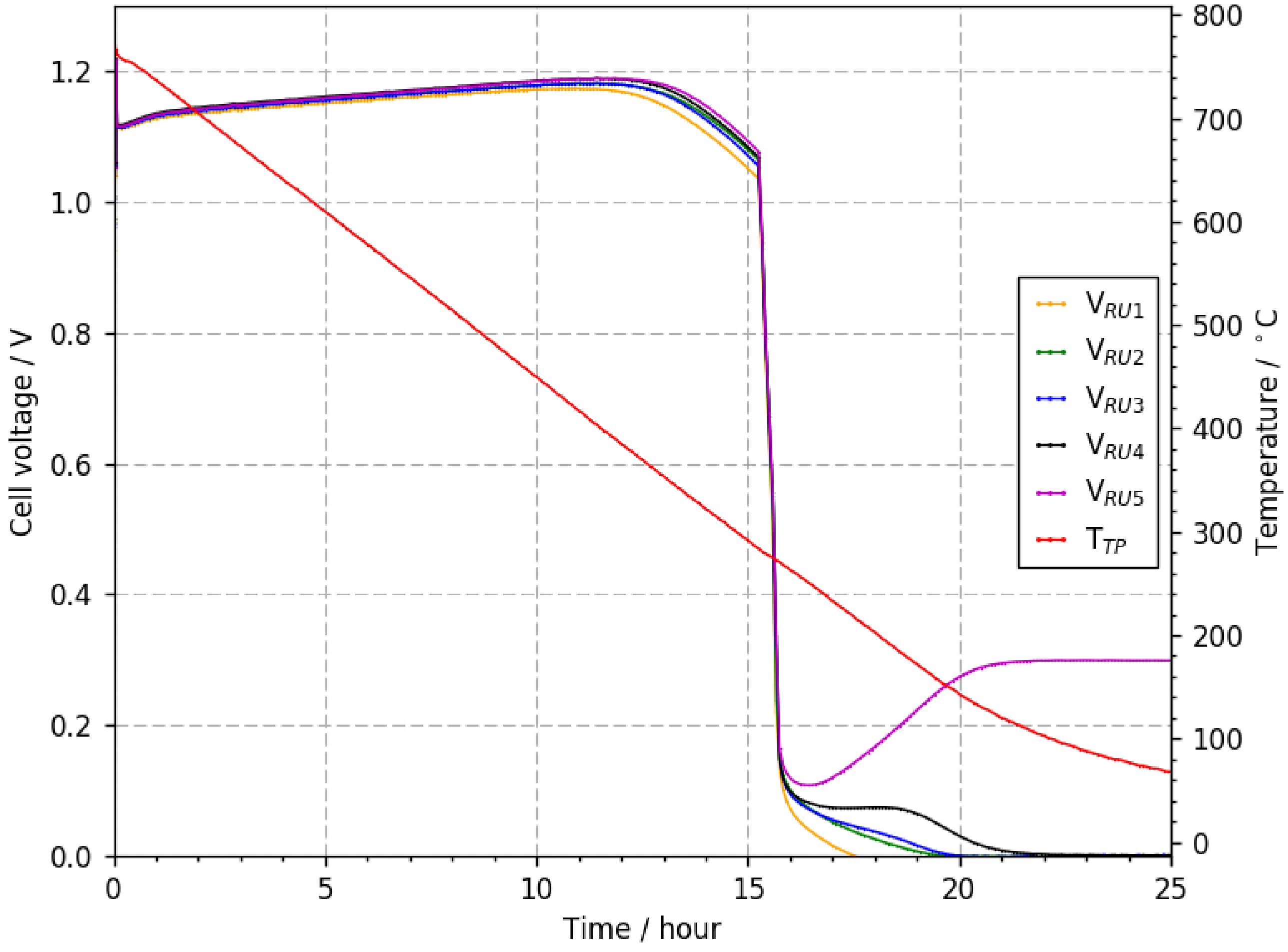

Figure 2: Stack temperature and RUs' voltage during shut-down of a 5 cells SOC stack test (Shut-downshut-down from SOFC conditions).

8 Differences to Existing Procedures

In general, the SOC shut-down procedure is provided by the stack manufacturer and therefore very limited existing procedures that has been published in this topic. Literature [1] only briefly describes the shut-down procedure. Nevertheless, this TM provides more detailed test steps with test input and test output parameters and data representation.

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