D5.1 – Report on test protocols for cell and stack test

Project information

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Contents

1. Abstract

The deliverable "D5.1 Report on test protocols for cell and stack test" presents the test protocols that will be used as guidelines for all the cell and stack test during the project. In the PilotSOEL project, cells with different processing methods, interdiffusion barrier layer, electrode microstructure and stacks with different components, assembly processes will be evaluated. It is thus important to be able to compare the performances of different cells and stacks, to evaluate the influences of those variations on the cell and stack performances and identify the key parameters for manufacturing high performing SOEL cells and stacks. Harmonized test protocols for both cell and stack are therefore made to ensure that all the cells and stacks are tested under same conditions or same selected conditions, so to ensure a better comparison of the test results.

2. Objective

The objective of the test protocol is to ensure a better comparison of cell and stack test results and to identify the key parameters (processing methods, inter-diffusion barrier layer materials, cell vs stack) for high performing and durable cell/stack manufacturing. For compiling the test protocols, current existing test protocols developed by JRC [1] and the previously EU-funded project SOCTESQA [2] were referred. The test protocol will be used in the cell tests activities in the PilotSOEL project as a reference and guideline.

3. Single cell test

3.1 Start up and fingerprint protocol

The startup protocol normally depends on the cell manufacture and the test station capability. In this project, the cells will be manufactured by DTU and ELCAS, respectively. For a good comparison of different manufacturing routes, it is recommended to reduce the cells at the same temperature, e.g. at 800 °C. During start up, nitrogen and then safety gas with 4 % H_2 in N_2 are normally supplied to the fuel electrode compartment and air is supplied to the oxygen electrode compartment. The fingerprint protocol includes different measurement points, at different temperatures as well as gas flow rates and gas compositions. Since this project only deals with H_2 production/steam electrolysis it is optional to run co-electrolysis conditions with carbon species feed streams. Air or oxygen or even no gas will be supplied to the oxygen electrode. The fingerprint will be characterized at 800, 750, 700 and 650 °C. The characterization methods applied for the fingerprint will be current-voltage (iV) curves and electrochemical impedance spectroscopy (EIS), cell voltage will be recorded all the time. The detailed measurement points and temperatures as well as gas flow rates and gas compositions are presented in Table 1. In case of durability characterized in other gas compositions, it is normally recommended to also include them in the fingerprint.

Table 1. Single cell startup and fingerprint protocol.

Points	T, °C	H_2, L/h	H_2O, L/h	Air, L/h	4 % H_2, L/h	N_2, L/h	Comment/measurement methods
1	25			20		20	Start up
2	800			20		20	Heat up
3	800			50	20		Reduction 2 h
4	650-800	23	1	50			H_2 + 4 % H_2O reduction 2 h
5	650-800	23	1	50			H_2 + 4 % H_2O, Air, iV, EIS
6	650-800	23	1				H_2 + 4 % H_2O, O_2, iV, EIS
7	650-800	19	5	100			H_2 + 20 % H_2O, Air, iV, EIS
8	650-800	19	5				H_2 + 20 % H_2O, O_2, iV, EIS
9	650-800	12	12	100			H_2 + 50 % H_2O, Air, iV, EIS
10	650-800	12	12				H_2 + 50 % H_2O, O_2, iV, EIS
11	650-800	1.4	12	100			H_2 + 90 % H_2O, Air, iV, EIS
12	650-800	1.4	12				H_2 + 90 % H_2O, O_2, iV, EIS
13	650-800						Gas composition at durability condition, iV, EIS@0A, EIS@-2 A

For iV characterization, a power supply will be used for controlling the current with defined steps, e.g., 1 A/min, and voltage will be recorded. A maximum current density (e.g. 2 A/cm^2), as well as upper (e.g. 1.3 V) and lower voltage limitations (e.g. 700 mV) will be predefined to avoid too harsh conditions applied on the cell during iV characterization as this may result in early degradation.

The EIS characterization will be performed at close to 0 A DC condition during fingerprint. Depending on the EIS instrument, a small DC current may be required.

3.2 Durability test

In the PilotSOEL project, the tests will be mostly performed under galvanostatic mode at ≤700 °C and electrolysis current density ≥0.85 A/cm^2. Reactant utilization will be considered when defining the gas flow rates or applied current. The test will be terminated if the cell voltage reaches above 1.6 V or the test time reaches 1000 hours. The fuel electrode gas composition shall be H_2 mixed with ≥50 % of H_2O, e.g., 10 % H_2 + 90 % H_2O, and the steam partial pressure can be varied for mitigation of the cell degradation, and the exact gas composition and flow rate will be coordinated with the stack test condition to ensure a direct comparison.

During operation cell voltage and cell temperature will be logged and the degradation will be evaluated based on the cell voltage and the operation time.

4. Stack test

4.1 Start up and fingerprint for stack test

The stack mounting and start up procedure for SOEL stack test is normally provided by the stack manufacturer. Stacks provided by ELCOY will be pre-reduced before delivery. Before startup, a leak test will be performed, and pressure drop on the stack will be checked and compared with Elcogen's stack manual. Furthermore, a short circuit check will also be performed with a multimeter to measure the resistance between voltage probes. During start up, a reducing gas composition of >4 % H_2 content is required when temperature is above 300 °C. The stack will be heated up to 650 °C at 2-3 °C/min. IV and EIS in SOFC mode will be performed to check the performance and compare with the performance measured by ELCOY during stack birth. For an ELCOY stack, the recommended operation temperature is below 700 °C. Detailed characterization points can be seen in Table 2.

Table 2. Stack startup and fingerprint protocol

Step	Temperature, °C	Fuel electrode gas	Air gas	Characterization
0	25	According to stack manual	According to stack manual	Leak test, checking the pressure drop on the stack at different flow rates. Short circuit check
1	25-620 (Furnace setpoint)	5 % H_2+N_2, 4.2 L/min	33 L/min	Voltages
2	620 (Furnace setpoint)	7.75 L/min H_2 +7.75 L/min N_2	33 L/min	SOFC-IV, +33 A EIS@ 2 A, 30 A
3	620 (Furnace setpoint)	5.2L/min H2 + 5.2L/min N_2	33 L/min	EIS@30A
4	620 (Furnace setpoint)	7.75L/min H2 +7.75L/min N_2	33 L/min	SOFC-IV, +33 A EIS@ 2 A
5	≤700 (Stack internal maximum)	7.75 L/min H_2 +7.75 L/min N_2	33 L/min	SOFC-IV, +33 A EIS@ 2 A
6	≤700 (Stack internal maximum)	H_2 + 20 % H_2O	25 L/min	SOEC-IV, -33 A EIS@ -2 A
7	≤700 (Stack internal maximum)	H_2 + 50 % H_2O	25 L/min	SOEC-IV, -33 A EIS@ -2 A
7	≤700 (Stack internal maximum)	H_2 + 80 % H_2O	25 L/min	SOEC-IV, -65 A EIS@ -2 A
8	≤700 (Stack internal maximum))	H_2 + 90 % H_2O	25 L/min	SOEC-IV, -65 A EIS@ -2 A
9	≤700 (Stack internal maximum)	H_2 + 90 % H_2O	25 L/min	Optional SOEC-IV, -105 A EIS@ -2 A
10	≤700 (Stack internal maximum)		Preferably keeping PO2<30% to limit risks of handling O_2 rich gases	Gas composition at durability condition, current shall not be below the targeted durability operation current. EIS@2 A

For iV characterization, a power supply will be used for controlling the current with defined steps, e.g., 1 A/min. A larger step can be adapted for decreasing the measurement time or minimizing the temperature gradient on the stack, and voltages of the repeating units and temperatures will be recorded. A maximum current density (e.g., 1 A/cm^2), as well as upper (e.g., 1.4 V) and lower voltage limitation (e.g., 700 mV) will be predefined to avoid too harsh conditions applied on the repeating unit during iV characterization as this may result in early degradation.

The EIS characterization will be performed at close to 0 A DC condition during fingerprint. Depending on the EIS instrument, a small DC current (e.g. 1 A DC) may be required.

4.2 Durability test

Durability test will be performed under galvanostatic (constant current) mode. In PilotSOEL, the targeted electrolysis current densities for durability test are ≥ 0.85 A/cm^2. The test will be terminated if one of the cell voltages reaches above 1.7 V or the test time reaches 2000 hours. Gas composition will be H_2 mixed with ≥50 % of H_2O, e.g., H_2 with 70 - 90 % H_2O. The steam partial pressure can be varied for mitigation of the cell degradation. The steam partial pressure for durability operation shall be coordinated with the cell test condition to ensure a direct comparison.

During operation cell voltage and cell temperature will be logged and the degradation will be evaluated based on the cell voltage and the operation time.

5. References

[1] T. Malkow, A. Pilenga, EU harmonised testing protocols for high-temperature steam electrolysis, JRC Publications Repository. (2023). https://doi.org/10.2760/31803.

[2] Final Report Summary - SOCTESQA (Solid Oxide Cell and Stack Testing, Safety and Quality Assurance) | FP7, CORDIS | European Commission. (n.d.). https://cordis.europa.eu/project/id/621245/reporting (accessed August 28, 2023).