AMMONIA TO POWER

D2.1 Testing protocol

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List of Abbreviations

Abbreviation	Definition
SOC	*Solid oxide cell*
SRU	*Single repeating unit*
SOFC	*Solid oxide Fuel Cell*
JRC	*Joint Research Centre*
IEC	*International Electrotechnical Commission*
SOCTESQA	*Solid Oxide Cell and Stack Testing, Safety and Quality Assurance*
OxiGEN	*Next-generation Solid Oxide Fuel Cell stack and hot box solution for small stationary applications*
VI	*Current Voltage*
EIS	*Electrochemical impedance spectroscopy*
ASR	*Area specific resistance*
EEC	*Electrical equivalent circuit*
KK	*Kramers-Kronig*
CNLS	*Complex non-linear least squares*
PFD	*Process flow diagram*
DOE	*Design of Experiment*
LSM	*Large stack module*
FBK	*Fondazione Bruno Kessler*
EPFL	*École polytechnique fédérale de Lausanne*
DTU	*Danmarks Tekniske Universitet*
VTT	*Teknologian tutkimuskeskus VTT*

01. Introduction

This deliverable is part of "Development of a next generation AMmONia FC system" project (AMON), which is funded by European Union in Horizon Europe through the work programme of the Clean Hydrogen Joint Undertaking (Clean H2 JU). AMON project aims at developing a novel system for the utilization and conversion of ammonia into electric power at high efficiency using a solid oxide fuel cell. AMON will promote the use of ammonia as a hydrogen carrier to enhance the flexibility of the energy system.

The scope of the work related to this report is establishing common procedures and test that will be conducted in the project. The foreseen tests have as target to investigate the behaviour of the cells and stacks when are fed with ammonia and evaluate the available performances and their degradation during time. The specific objectives of the project and call topic are summarized below.

Moreover, the possibility to test different testing platforms (single SRU, short stack, stack, and full stack), make available to perform an optimization of design of experiment to maximize test output.

The report is divided as follows. In the second chapter, it summarizes the objectives of the project and of the experimental activities. The following chapter briefly reviews the testing protocols in literature and recaps the principal experimental result of direct ammonia solid oxide fuel cell systems. The chapter four is the core of the work. First it defines the test modules representing the common ground of the different experimental activities. Then, different thermal stabilization criteria are presented. Finally, the design of experiment defines the testing protocols. The last chapter resumes and assigns the different test to different test platforms.

02. Objectives

Objective of testing activity is to demonstrate the achievement of project's target with dedicated experimental activity on different test platforms. The main targets are:

- O1: Target 70% system electric efficiency.
- O2: Validate the stack and system flexibility to allow for 30% partial load and full flexibility in the overall range.
- O3: Qualify the system for at least 3000 hrs operation.
- O4: Demonstrate 90% availability in the operating hour.
- O5: Demonstrate less than 3% degradation rate at nominal power condition measured over 1000 hours of continuous operation.

Additional objectives of test activity are:

- O6: support the definition of control strategy (operative state, transient operation, setpoint and gradient for final system).
- O7: Impact of recirculation on pressure management on the stack and support to the definition of pressure control loop
- O8: evaluate gas crossover between cathode and anode side and vice versa for the support to the safety assessment.
- O9: evaluate the effect of ammonia usage on cell and stack components (corrosion, nitrification, etc.)

03. Review of test protocol

This chapter presents a short revision of testing protocols from IEC and JRC, and previous European projects on SOFC. Unfortunately, no testing modules or standards are available for solid oxide cell fed by ammonia. Furthermore, due to this lack of information, a summary of the test available in the literature about ammonia SOFC is provided.

The main testing protocol available in standard and scientific literature can be summarized as follows:

- IEC 62282-7-2[1] and IEC 62282-8-101: are the only two international standard on the stationary SOFC testing, providing guidance on the different type of test, testing definition and reporting. The standard includes characterization tests like current voltage characterizations, effective fuel utilization test, internal reforming performance (for methane). Also, endurance tests are explained in terms of durability test and thermal cycling. At deeper level in the characterization Electrochemical impedance spectroscopy and current interruption method are considered to evaluate resistance/impedance characteristics. About conditioning and stability criteria no particular information were supplied, because the standard assumes that this information should be provided by the SOC manufacturer.
- JRC SOFC test modules[2]–[5]: in this case four test modules are available, on single cell or stack and on polarization curves or endurance tests. In this case, also diesel is considered as a fuel together with hydrogen or methane. Indication on the conditioning and stability criteria are provided and will be discussed in the paragraphs of the test modules. The polarization curve characterization envisages two approaches sweeping or stepping the current. On the endurance test, for the single cell are presented two kinds of test with light or heavy duty, meaning in different percentages of fuel utilization and a different percentage of utilized fuel at anode inlet. In these cases, the fuel composition at the inlet is prescribed.

Additional test protocols for SOFC are present in different European project.

- The most prolific of these is SOCTESQA that aimed to harmonize the testing of SOFC/SOEC[6]. Several testing modules are available, from general considerations of the SOC testing to EIS and endurance analysis. Particular attention was paid on the conditioning and stability criteria.
- Another project that presented a test protocol is the Insight[7] project, which focused on monitoring and diagnostic of SOFC systems. In the deliverable D2.1, two test module on durability test are provided together with fault modules (fuel starvation, gas leakages and carbon poisoning).
- The deliverable D6.1 of the OxiGEN[8] was focused on establishing common testing criteria for the benchmark of different solid oxide cells. In the report, VI and EIS test conditions are described, which are included and repeated inside a long-term testing (500h). Furthermore, in the tests, heat treatments, like sealing and reduction, are comprised.

As mentioned above, to the best of authors knowledge in literature specific test protocols for ammonia-fed fuel cell are not present. Because of that, in the appendices the results of tests of ammonia SOFC from scientific literature are reported with performance test and long-term tests. In some of the entries are also reported the performances obtained employing pure H2 as fuel. This summary of tests, which doesn’t pretend to be complete, could a useful base for the development of test protocols, and their parameters.

04. Test protocol definition

In the section, test protocols to investigate direct ammonia solid oxide fuel cell performance are developed and described based on the state of art of testing activity in scientific literature and from standard or other guidelines and reports. Test protocols are defined as consequential flow of different test model, organized as block-structured programming, expressed as single test modules, repeated in specific way to build up complete test protocol. In this sense:

- Test modules

Test modules represent the single test action (leakage test, linear sweep, etc) including a description about what in-operation action are performed, what is the exit criteria and available range of parameters. They are basically single, independent step of testing. Amount them it is possible to include starting up operation, conditionings, state of Hot standby, current voltage characteristics, etc. All single modules are reported in next section.

- Test protocol

Test protocol defines the procedure and method to address the investigation and verification of a unique or a series of targets. They are developed as sequences of test modules, concatenated with dedicated starting criteria, exit criteria, choice selectors and terminal criteria, aiming to perform sweep parameter's investigation.

A fundamental aspect of the test modules and protocols is the criterium to define the pseudo-equilibrium state of system is achieved to usefully gather the data. The specific challenge of solid oxide fuel cell operating at high temperature and with relative high time constant for heat diffusion in cell/stack[1], requires the necessity to define a dedicate criterium to identify equilibrium state/condition. Without this, data collection and results can be affected by relevant systematic errors, with losses of reproducibility and repeatability of data between different research teams involved in the activity. Several approaches are reported in the next chapter.

1.1. 4.1 Test modules

This paragraph introduces the principal test modules with a brief description and the proposed test included. The main parameters considered are presented in Table 1.

Table 1 - Parameters included in the test protocols with their symbols

Symbol	Definition	Unit	Dependent/ Independent
j	*Current Density*	*A/cm²*	
I	*Current*	*A*	
V	*Voltage*	*V*	
T	*Temperature*	*K*	
c_p	*Specific heat capacity at constant pressure*	*J/K*kg*	
m	*Mass of the stack*	*kg*	
A_s	*Heat exchange area*	*cm2*	
h	*Heat transfer coefficient*	*J/K*m²*	
f	*Gas flow rates*	*Nl/min*	
P	*Power*	*kW*	

[1] Time constant means the ratio between thermal capacity of cell/stack and heat exchange capacity ofanode/cathode.

F	*Faraday constant*	*C/mol*
U_f	*Fuel utilization*	-
A_{cell}	*Cell area*	*cm^2*
n	*Number of cells in the stack electrically connected in series*	-
t	*Time*	*h*
p	*Pressure*	*mBar*
U_{ox}	*Oxidant utilization*	-
n_{cycles}	*Number of cylces*	-
T_{op}	*Operative temperature*	*K*
T_{down}	*Lowest temperature of the cycle*	*K*
$\Delta T/\Delta t$	*Heating/cooling rate*	*K/min*
V_{H2}	*Volumetric flow rate of fuel*	*Nl/min*cm^2*
V_{air}	*Volumetric flow rate of air*	*Nl/min*cm^2*

Leakage Test

The leakage test module represents an essential requirement for the correct operation of the SOFC. The test is usually conducted at ambient temperature and repeated in operative condition (above 873K). Leakages over a threshold compromise the performances of the cell/stack and may lead to unpredicted leakage of ammonia.

The test is directly conducted by Solydera for the cell, the SRU, the stack and the stackbox.

It is suggested to develop a leakage test to verify the stack or stackbox connection with the test bench, especially for the manifold flange connection. For the correct procedure bring the system to 30 mbar by closing or partially closing the fuel and air outlets of the system and circulating forming gas and air. With a sniffer verify all the critical points such as flanges and connections. The leakage detection should be lower than 50 ppm. If higher try to tighten the connections.

Start-up

Due the sensitive nature of the solid oxide stacks and their operational temperature, the heating procedure should be conducted carefully and with aligned parameters (setpoint, gradient, flows, etc.). To preserve the cells from damages above a certain temperature level a conditioning in term of reductive gas flow needs to be provided (i.e., H2N2 gas flow).

Procedure and parameters

*Heating procedure (by SolyDera): from ambient temperature to 873.15K following a heating ramp with a maximum temperature gradient equal to 2 K/min. Forming gas (95% Nitrogen- 5% Hydrogen, not flammable mixture) uses with a flow rate equal to 2.86 Nml/(min*cm2) and air with a flow rate equal to 66.43 Nml/(min*cm2).*

Over 873.15K to standby conditions, the same flow rate for the benchmark curves can be applied.

Start-up condition is considered as concluded when cell/stack is in thermal equilibrium at the decided standby condition (Temperatures, flows and composition).

Conditioning

Conditioning is a test module introduced in several reference for SOFC test. Here, the cell/stack is operated at constant duty until the voltage and temperatures reach a stable condition. The conditioning step is usually executed after the start-up of the SOC and just before the start of the real testing phase (voltage-current, or others). An example of constant duty could be the SOC operating in SOFC mode at partial load (0.2 A/cm2).

Exit condition for such test module is the achievement of an equilibrium state in terms of voltage and temperature.

Current voltage characteristics

Polarization curves (Stack/cell voltage versus current or current density) are one of the most employed basic methods to characterize the performances of Solid Oxide stack or cell, in terms of density of current and power. The results obtained by this test are mainly the variation of voltage as a function of the current density. An important parameter that could be derived from the VI test is the area specific resistance (ASR)[2]. However, the voltage dependence cannot be reduced to current density, other important parameters are current variation rate, gas flow rates, gas compositions and stack temperatures[3].

The procedure of these test can be summarized as follows:

1. *Set up temperature of the stack (cell)*
2. *Set the gas flow rates.*
3. *Wait until the thermal stabilization (see 1.2) is achieved.*
4. *Start the test with a constant current ramp or a current step with a proper stabilization time decided as below .*
5. *The test finishes when both conditions are achieved:*
    a. *the fuel utilization reaches 85%.*
    b. *Thermal equilibrium (see 1.2) is achieved.*

The exit criteria to preserve the cell functionality is achieved setting a breakout voltage of 0.68V/cell and 0.7V as readjust voltage, if this is not possible the breakout voltage is set to 0.7V/cell.

Electrochemical impedance spectroscopy

The other characteristics of the cell that can be useful for an electrical equivalent circuit (EEC) model considering not only Ohmic resistance but impedance including different resistance and capacitance, can be investigated by employing electrochemical impedance spectroscopy. From the fuel cell's[4] impedance spectrum is possible to get information, throughout plots (like Nyquilst or Bode), about the reaction kinetics, ohmic conduction processes, mass transport and other properties by parameter identification, for example, by complex non-linear least squares (CNLS) fitting of the measured (and Kramers-Kronig (KK) validated) EIS data to the selected EEC model. This test module is specific for single cell test platforms; however, it could be also applied on stacks, but the test benches included in the project cannot perform EIS test on stacks.

The procedure of these tests can be summarized as follows:

1. *Set up temperature of the cell.*
2. *Set the gas flow rates.*
3. *Set voltage or current baseline.*
4. *Wait until the thermal stabilization (see 1.2) is achieved.*
5. *Start EIS measure with specific parameters about:*
6. *Range(s) of perturbation frequencies (100mHz-1MHz as well established in literature[10]–[12])*
7. *Amplitude of signal stimulus*
    a. *Number of samples per each excitation frequency*
    b. *Number of measurements for each frequency decade*

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[2] Defined as: total resistivity of a cell or stack in operation, including the change of voltage (potential) due to one or more electrochemical reactions [9].
[3] Defined as the average temperature between the inlet and outlet air temperature.
[4] Only single cells will be tested employing EIS in the project.

c. *The test finish EIS procedure is finalized.*

Current interruption

Current interrupt method is another electrical characterization technique that can distinguish between ohmic and nonohmic fuel cell processes. In this test, an interruption of the applied current induced a variation of voltage, the time independent variation is due to the ohmic resistance, and, in the other case, the slower variation can be identified with non-ohmic resistance. This technique will not applied during the project test because the EIS test will be applied at single cell level.

Procedure and parameters:

After the proper conditioning and stabilization time the test can start. The test can be conducted within a polarization curve with time stepping and interrupting per every point in the VI curve to estimate properly the area specific resistance of the SOC.

Benchmark test (H2)

This test module refers to the test usually driven by SOLYDERA to check the performance of the cell. The comparison between the values provided by the manufacturer and the ones obtained in the test modules help to understand the state of health of the SOFC and if the performances are equal to the expected ones.

Procedure and parameters:

*Set the hydrogen flow rate equal to 3.57 Nml/(min*cm2), the nitrogen flow rate equal to 2.32 Nml/(min*cm2) and the air flow rate equal to 66.43 Nml/(min*cm2). Bring the outlet air temperature equal to 1023.15K. Develop a polarization curve till reaching Uf=85%.*

Shut down

Similarly, to the heating module, the shutdown should be conducted carefully, avoiding thermal stresses of the SOC.

Procedure:

The Cooling procedure is performed by high temperature condition (without load applied). Above 873.15K, the flow rates replicate the benchmark test ones. Below 873.15K, the cooling rate must be lower than 2K/min with a reducing gas flowrate equal 2.86Nml/min until near ambient temperature is reached.

1.2. 4.2 Thermal stabilization criteria

The stabilization of the cell/stack is the essential step to assess the influence of operating conditions on the system, thus a stability criterion is required. In this regard, usually the stable condition of a parameter is reached if a threshold is not exceeded by the parameter variation during a time range. The choice of the threshold and the time range requires several considerations in terms of the measurement capacity of testing facility, overall test duration, dimension of the tested SOC. Here, three different approaches are reported.

In the project SOCTESQA, it was proposed a general stability criterion giving a starting point for specific stability criteria. Table 2 reports the proposed thresholds for the different parameters.

Table 2- Proposed thresholds for SOC parameters from SOCTESQA project

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
$f_{neg/pos, in/out}$[5]	1 Hz	3 min	3% FS [6]
$p_{neg/pos, in/out}$ Errore. Il segnalibro non è definito.	1 Hz	3 min	3% FS

Another stability criterion was proposed in the “Test module” from JRC where the waiting time is stated in 10 minutes and a voltage variation below +/-5mV (with a sampling time of 5min); in the same module the conditioning step is set to last 20h minimum.

A more robust approach can be derived by the time constant of heat transfer in the cell/stack and the uncertainty of the measures. This method is proposed herein. Due to the different scale of test that are performed in AMON project, from single cell to full stack (8kWel), a relative approach that depends on the scale of the SOC tested can be proposed here. The thresholds may be set as the measurement uncertainty from the rolling average of the parameter for the half of sampling time. The sampling time and the hold time can be set evaluating the time constant of energy balance of the system in terms of heat transfer. Assuming the limit of control volume at the SOC casing without the insulation layer, in a first approximation the system can be evaluated in a lumped capacitance model, with an energy generation in the cell, the heat exchange between SOC (cells) and gases, and the heat storage of the SOC. It results in a time constant:

$$t_c = \frac{c_p \cdot m}{h \cdot A_s}$$

Where h is the convection heat transfer coefficient for the cell and As the SOC arealthe total active area), cp the specific heat capacity at constant pressure and m the mass of the stack SOC. Frome the time constant, the holding and sampling time can be set as from 3 to 5 times larger than the calculated value.

For the criterion to evaluate the variation threshold for any parameter and, therefore, the related stability, it can be considered as stable if the parameter presents a variation lower than the measurement uncertainty (three times the standard deviation) during the first half of the sampling time.

1.3.4.3 DOE – Design of Experiment

This paragraph starts reviewing the main limitations and boundaries of test parameters due to the testing facilities and SOC cells/stacks capabilities. Then it deals with testing protocols and procedures of the tests on the SOC cells employing the previously presented test modules.

In the definition of the testing input parameters in the foreseen tests, the limitation and constraints coming from the SOC cells/stacks will create the base for the definition of the testing protocol.

The principal limitations in parameters for the different SOC scales are summarized herein. In general, it is suggested not to overcome an Uf value higher than 85% (related to H2 from ammonia cracking). For the oxygen don’t overcome Uox=25%. The suggested value for air flow rate 50Nml/min cm2 that will be employed in all tests reported herein.

The suggested temperature range is 923.15--1073.15K. Below the performance could be too low especially with ammonia, above the sealing glass material could be damaged. A maximum ramp rate of

[5] Neg/Pos: referred to the positive and negative electrode; In/out: inlet and outlet of cell/stack
[6] Full Scale: referred to the maximum value allowed by the flow meter.

2 K/min is suggested. For the stack and stackbox pay attention not to have a temperature difference between the flow in and out higher than 373.15K otherwise the thermal stress on the stack would be too high.

Reactant utilization

Reactant utilization refers to the test module evaluating the SOFC performances as function of reactant stoichiometry. In other words, the polarization curve is done with different fuel and air utilization, helping to investigate possible limitations of gas distribution and transport.

Procedure and parameters

After the proper conditioning and stabilization time the test can start. The test will repeat the current voltage characterization test module, in this case the polarization curve is conducted with the same fuel utilization, therefore the mass flowrate is changed with the current steps of the polarization curve. In Figure 1, the flow diagram of the complete test is reported.

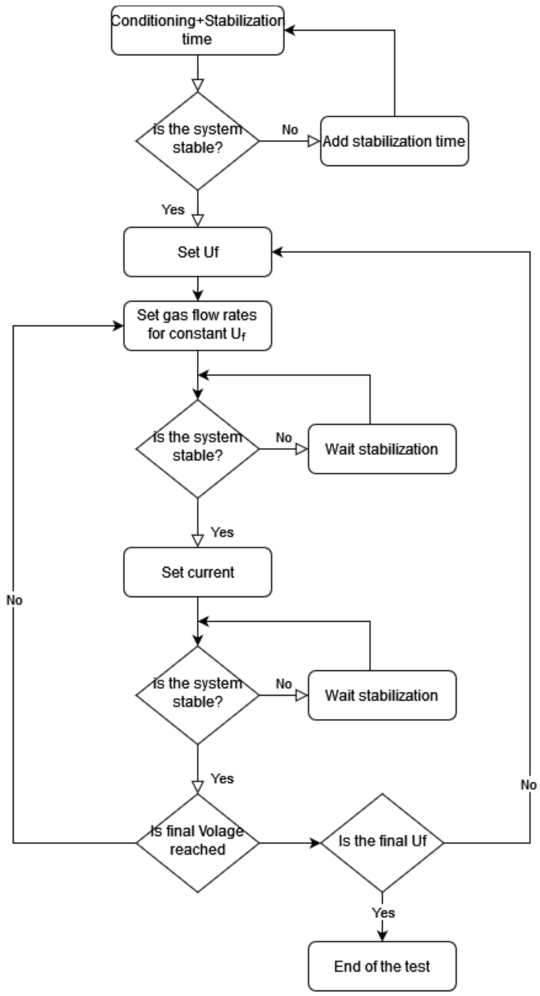

Figure 1- Reactant utilization test flow diagram

The test will evaluate the influence of fuel utilization up to 85% with different current densities/load (from 0.1 to 0.4A/cm2). The start of the test will set the fuel flow rate calculated with

$$V_{H_2} = \frac{I \cdot n}{U_f \cdot F \cdot 2 \cdot A}\ 22.461 \cdot 60\ [\frac{Nl}{min \cdot cm^2}]$$

Where the symbols are referred to respectively: I to the current, n to the number of cells, F to the Faraday constant, A to cell area and Uf to fuel utilization. The fuel utilization is set as first parameter, then the different flowrates are calculated for all the steps of current until the maximum current density.

On the air utilization, the flow rate can be calculated with

$$V_{Air} = \frac{I}{U_{Ox} \cdot 4F \cdot A} \cdot \frac{100}{21} \cdot 22.461 \cdot 60 \, [\frac{Nl}{min \cdot cm^2}]$$

The maximum air utilization should not exceed 25%.

Reactant gas composition (cracking/recirculation)

This test module envisages the evaluation of SOFC as a function of the reactant type at feed of the SOFC. In particular, for the AMON project, the fuel gases can be pure H2 or NH3, H2/N2 and H2/N2/NH3 blends, varying gas compositions it is possible to simulate the percentage of external cracking or the recirculation system.

Procedure and parameters:

Also in this case the procedure will repeat the VI characterization, with different gas compositions. The proposal in this case is to analyze the effect of the external cracking, the recirculation separately and then with a combination of them. The flow diagram is presented in Figure 2.

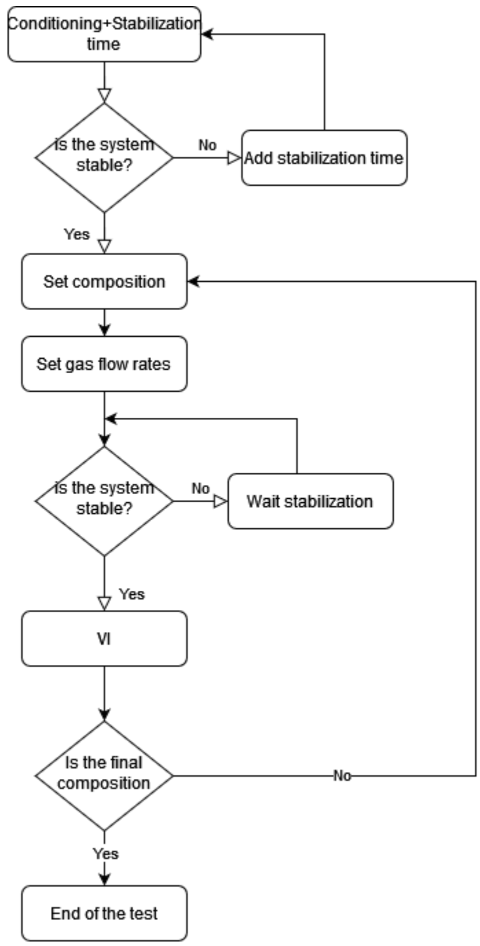

Figure 2- Reactant gas composition tests flow diagram

For the external cracking effect on the SOC performance the procedure includes three level of ammonia flow directly feed to cells for example 10, 20 and 30% of the total flow rate.

For the recirculation, five levels could be evaluated from 50 to 90% with 10% step.

For the combination of them, it will include all the recirculation steps and two levels of external cracking: 10%, equal to the proposed PFD for the final prototype, and 30%.

Temperature sensitivity

The SOFC performances as a function of the temperature are the characteristics investigated in this test modules. The stack operating temperature is generally varied with the variation of gas inlet temperature (especially sweep) and the oven temperature.

Procedure and parameters:

In this module, the test will apply the previous VI characterizations (fuel utilization and reactant compilation) to investigate the influence of stack temperature on the performances. The proposed range is between 948.15 and 1023.15K with 25K step.

The Figure 3 presents the related flow diagram.

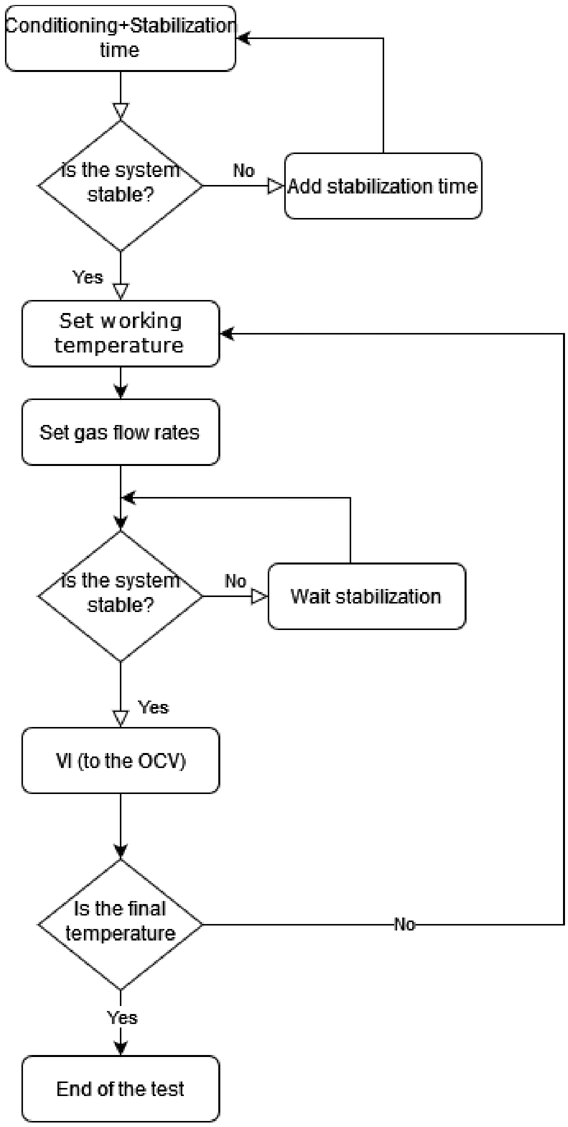

Figure 3- Flow diagram of temperature sensitivity test

Operation under varying current

The variation of load with time is usually required in many applications, thus it is important to evaluate the dynamic operation of the stack moving from partial to full load or the contrary. The load profile will be defined depending on the chosen applications. One of the objectives of the project imposes a minimum load of 30%.

Procedure and parameters:

The test will include different VI ramps starting from some partial load levels (30-100%). The exact sequence should be evaluated considering in which the application the ammonia fed SOFC will be employed.

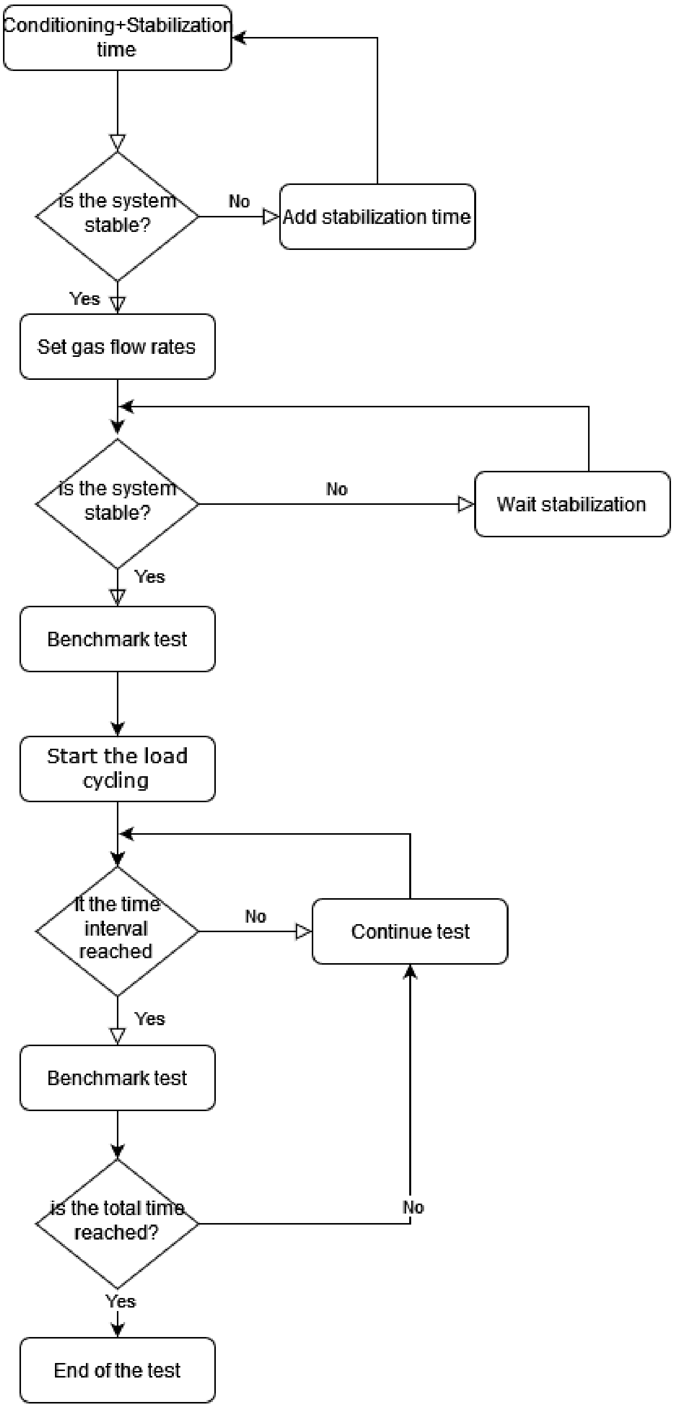

Figure 4- Flow chart of load cycling test

Long term test (durability)

The analysis of the degradation of the SOFC over time can be conducted in an endurance test under steady-state conditions. Usually, these tests are operated in galvanostatic mode (constant current), with the output expressed in terms of voltage and power (this is calculated). The degradation is evaluated by the power decrease over time. Another important parameter that comes out from this type of test is the stack's drifting temperature where the oven and gas temperatures in input are maintained constant. The degradation rate can be defined as[13]

$$DR = \left|\frac{V(i,t) - V(i,t=0)}{V(i,t=0)}\right| \times \frac{1000}{t} \times 100\%$$

Where V(i,t=0) is the voltage at the starting time at the prescribed current density condition.

To evaluate the impact of partial load on durability, long term tests at the minimum load could be performed.

The Figure 5 report the flow diagram of the long-term test.

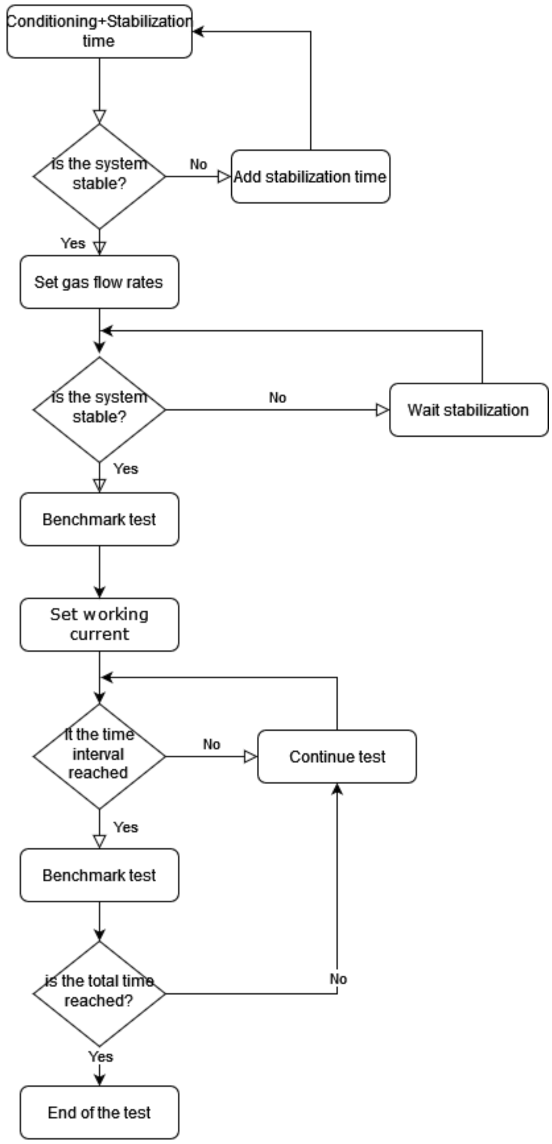

Figure 5- Long term test procedure reduced to flow diagram

Thermal cycling

This test evaluates the durability of the SOC with a thermal cycling. The cycle can be divided in four steps: performance test (at op. Temperature), cooling, waiting at minimum temp and heating up to operating temperature. In this test several parameters should be set such as number of cycles, operating and cooled temperature, heating and cooling rates, waiting time.

Table 3- Input parameters for thermal cycling

Parameter	Definition	Proposed value
n_{cycles}	number of cycles	1000
T_{down}	low temperature of the cycle	373.15, 723.15K (full and half cycle)
T_{op}	operating temperature (VI test)	973.15,998.15K
ΔT/Δt	heating cooling rate	Lower than 2K/min
t_w	Waiting time in Top and Tdown	Equal to double of the stabilization time

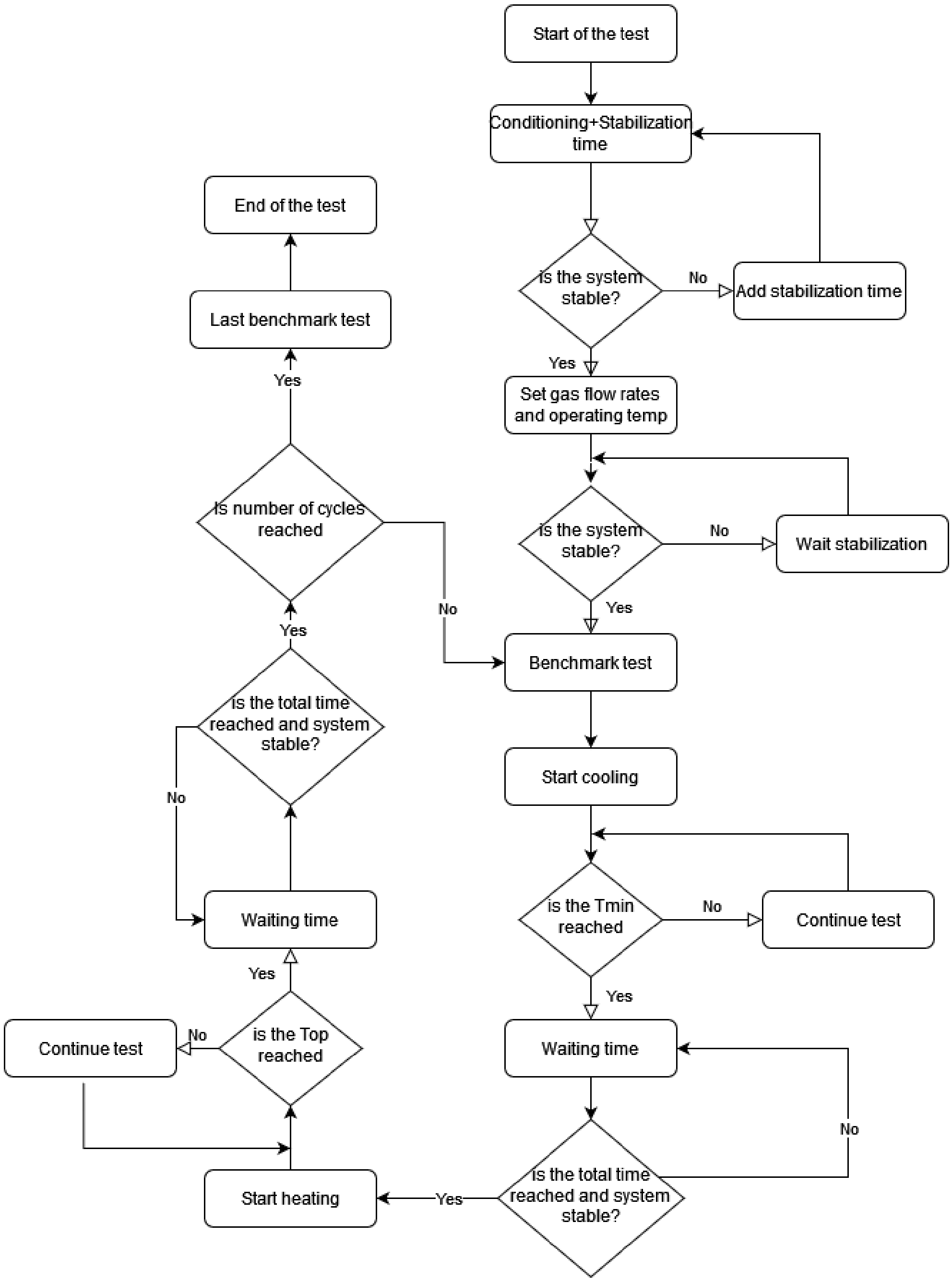

Figure 6 - Diagram for the thermal cycling test

Accelerated tests

Another way to characterize the degradation of fuel cells is employing accelerated tests, which can evaluate durability in a short period of time. In such kind of tests, operating conditions outside the usual operation windows are applied to accelerate degradation mechanism on material and components, similarly as actual service conditions but at faster rate. As a result, the testing time needed is reduced.

In the JRC technical report[2], Tsotridis and Pilenga explained the differences between Accelerated Stress Test (AST) and Accelerated Life Test (ALT). The AST is normally employed to help develop new materials; thus, it is applied to reproduce selective degradation on specific components or materials. The latter kind is useful to investigate durability of the fuel cell aiming at mimicking the failure during real world degradation.

Both employs stressor operating conditions to increase the degradation rate if compared with reference operating conditions. The tests apply dynamic loading to the cell, regularly analyzing the state of performances of cell/stacks. Stressors are defined as input parameters that are deviating from reference conditions, which are impacting on the performances and durability of the cells/stacks and causing deviation from normal operation.

Similar to the load cycling test, in the accelerated test it is needed to provide a dynamic profile.

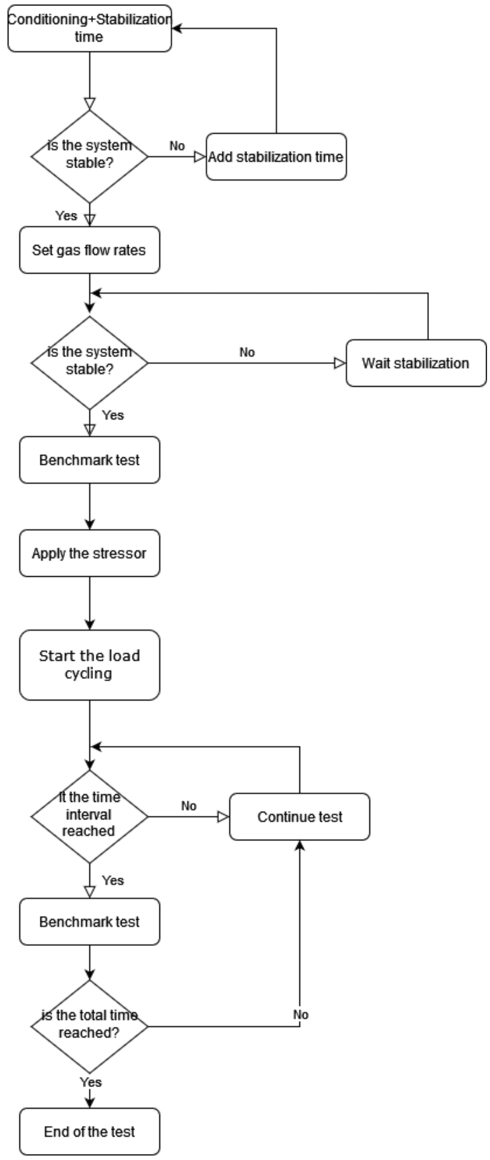

Figure 7 - Flow chart of accelerated test.

The only stressor identified is the type of fuel (Ammonia percentage), evaluating the impact of fuel composition variation on the stack degradation. The nominal value is set to 10% in volume as defined preliminary analysis of the work package 1.

Table 4-– List of stressors with their nominal, maximum and minimum values

Stressor	Nominal Value	Maximum value	Minimum value
Fuel Composition (NH3%)	10%	100%	0%

Post-Mortem analysis

During post-operation analysis, the stacks are disassembled to look at the macroscopic changes that occurred during the operation. The variations regard both the stack structure and the cells.

Particular attention is focused on the ammonia effect on the steel and cell components by looking at nitridation phenomena. Samples of cells and stack components are collected for further microstructural analyses.

Variables and range

In the following table, the main parameters set for the different DOEs are summarized. For all the test an air flow of 50 Nml/min*cm2 is suggested as initial guess value in order to have a proper thermal stabilization.

Table 5- Main parameters for the DOEs

TEST	TEST INPUT PARAMETERS	RANGE	STEP
Reactant Composition	NH_3	100% -10%	15%
	H_2-N_2	Composition: 75:25 to 30:70	Depend on the recirculation level
		Total volume fraction: 0-90%	15%
	Recirculation level	0-90%	15%
Reactant utilization	Fuel	Up to 85%	Continuous in VI
	Air	Up to 25% (Suggested 12.5%)	-
Temperature sensitivity	$T_{cell/stack}$	*973.15, 1023.15K*	25K
Operation under varying current	Load variation rate (% of nominal power/min)	3 A/min	-

	Number of cycles	>=100	-
Long-term test	Hours	1000h @0.4A/cm^2	-
Thermal cycling	T_{min}	100-450°C (Full or half cycle)	
	T_{op}	973.15, 1023.15K	25K
	Number of cycles	25	
	Temperature ramp	Up 2K/min	
AST	Stressor (NH3 %)	0-100%	10%
	Number of cycles	100	-

05. Test platform

In this paragraph, the different tests carried out by the different platforms will be summarised in the following table. As explained earlier in the document, some tests will be repeated in the different platforms, others are specifically related to the specific platform, and all aim at the objectives defined in section **Errore. L'origine riferimento non è stata trovata.**.

Table 6- Tests and objectives assigned on the different test platforms.

<table>
<tr><th>Test Platform</th><th>Partner involved</th><th>Objectives (section Errore. L'origine riferimento non è stata trovata.)</th><th>Test Protocol</th></tr>
<tr><td>Single cell</td><td>EPFL</td><td>01/09</td><td>EIS, postmortem analysis</td></tr>
<tr><td colspan="4">Single-cell platform tests the single-cell material (cermet including anode, cathode, and electrolyte) for the AMON project. The characterization activity investigates the performance of cell fed directly by ammonia and with simulated composition in order to verify the efficiency and to evaluate the ammonia effect in post-mortem analysis of cell material.</td></tr>
<tr><td>Single Cell/SRU</td><td>DTU</td><td>01/09</td><td>EIS, postmortem analysis</td></tr>
<tr><td colspan="4">SRU platform tests the single cell package (cermet including anode, cathode, and electrolyte and GDLs and sealing) for the AMON project. The characterization activity investigates the performance of cells fed directly by ammonia and with simulated composition to verify the efficiency and to evaluate the ammonia effect in post-mortem analysis of cell structure.</td></tr>
<tr><td>Short stack (6 cells)</td><td>FBK</td><td>01/02/03/04/05/08</td><td>Reactant composition, long term test, Temperature cycling, AST, load cycling, Reactant utilization</td></tr>
<tr><td colspan="4">Short stack platform tests a 6-cell for the AMON project. The characterization activity investigates the performance of a short stack fed directly by ammonia and with simulated composition to verify the efficiency and to evaluate the ammonia effect in post-mortem analysis of cell structure. Additional testing activity will operate temperature and load cycling to stress the short stack to evaluate resistance and resilience under a rapid gradient of the stressor (current and temperature). A long-term test and AST procedure are included to evaluate the effect of ammonia on cells for several hours.</td></tr>
<tr><td>G8-80</td><td>FBK</td><td>01/02/03/04/05/06/07/08</td><td>Long term test, load cycling, testing control strategy (custom test protocol)</td></tr>
<tr><td colspan="4">G8-80 platform tests a 70-cell stack(80cm2) for the AMON project. The characterization activity investigates the performance of stack fed directly by ammonia and with simulated composition, to verify the efficiency and to evaluate ammonia effect in post-mortem analysis of stack structure. Additional testing activity will operate load cycling to stress the stack to evaluate resistance and resilience under a rapid gradient of the stressor (current) and to verify a dedicated control strategy for transients. A long-term test procedure is included to evaluate the effect of ammonia on the stack for several hours.</td></tr>
<tr><td>G8-X</td><td>VTT</td><td>01/02/03/04/05/06/07/08</td><td>Operation modes, long term test</td></tr>
<tr><td colspan="4">G8-X platform tests an 80-cell stack (320cm2) for the AMON project. The characterization activity investigates the output of a large stack fed directly by ammonia and with simulated composition to verify the electrical performance in a large stack. Additional testing activity will operate in a controlled environment, and a specific test protocol will be defined once the operative modes of the AMON prototype are defined. The scope of this activity is to check the control strategy for the AMON prototype regarding the operation modes and transition. A long-term test procedure is included to evaluate the effect of ammonia on the stack for several hours.</td></tr>
<tr><td>Final DEMO</td><td>SAPIO</td><td>01/02/03/04/05/06/07/08</td><td>Operation modes, long term test</td></tr>
<tr><td colspan="4">Final demo plant including G8-X stack and dedicated BoP for the AMON project. The demo plant will be tested for a cumulative time of more than 3000H and 1000h of continuous operation.</td></tr>
</table>

06. Conclusion

The aim of this report was the definition of common testing protocols for Ammonia SOFC of the different test platforms included in the AMON project.

In the first chapter, the report resumes the Amon project and illustrates the report content.

In the second chapter, main objectives of AMON project are exposed. These objects are the driver for the choice and tuning of dedicate test modules and so protocols.

In the third part, a short overview of experimental test objectives was carried out. Then, a brief review of testing protocols and results achieved in the literature are presented. It should be noted that to the best of the author's knowledge, at the present time, no specific test for ammonia fed SOFC is available in standards or harmonized procedures. Most of the test results available in the literature evaluate VI performance with different flow rates and fuel utilization. Only a few studies report long-term tests with more than 500h.

In Chapter 4, the deliverable defines the test protocol starting from the definition of test modules, which are the constituent elements of the testing protocol. The test modules define the procedure for fundamental test like the polarization curves that are then employed in the test protocols. Following the test modules, the report examinates different thermal stabilization criteria and proposes a new approach that considers the multiple sizes of the test platform considered in the project. Finally, the test protocols and the related design of experiment are presented.

In chapter 5, the distribution of test protocols and role for the characterization of Direct ammonia fed SOFC. Is reported and assigned on different test's platforms (form cell, to LSM).

Then, the report reviews the testing platforms and defines the test applied to them. For every platform, it evaluates the objectives of the test and the test included.

AMMONIA TO POWER

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Appendix

In this appendix, the result of a brief review of the performances for ammonia fed SOC that are available in scientific literature. The Table 7 report the maximum performances obtained by current voltage curves with their temperature and compared, when available, with pure hydrogen. The other table, instead, summarised the results of long term tests with degradation.

Table 7- Principal results of experimental test for ammonia fed SOFC

Cell	Support	Electrolyte (AN/EL/CAT)	Temp. (°C)	Performance (mW/cm^2)		Fuel composition		Ref.
				Ammonia	Hydrogen	Ammonia fuel[7]	Hydrogen fuel[7]	
Planar	Electrolyte	Ni-YSZ//YSZ//Ag	800	75	64	100%	100%	[14]
		Ni-SDC//LSGM//Pt	700	10	13	100%	100%	[15]
			800	27	90			
			900	120	260			
		Fe-SDC//LSGM//Pt	700	73	102	100%	100%	
			800	151	171			
			900	242	291			
		Co-SDC//LSGM//Pt	700	9	13	100%	100%	
			800	24	44			
			900	85	120			
		Ni(40)Fe(60)-SDC//LSGM//SSC	700	165	…	100%	–	
			800	254	–			
			900	360	–			
		Ni-SDC//LSGM//SSC	700	36	–	100%	–	
			800	118	–			
			900	253	–			
		Ni(97.5)Mo(2.5)-SDC//LSGM//SSC	700	106	–	100%	–	[16]
			800	290	–			
			900	416	–			
		Ni(97)Ta(3)-SDC//LSGM//SSC	700	29	–	100%	–	
			800	216	–			
			900	322	–			
		Ni(97)W(3)-SDC//LSGM//SSC	700	24	–	100%	–	
			800	161	–			
			900	313	–			
		Ni (97) Cr (3)-SDC//LSGM//SSC	500	25	58	50% NH_3, 50% N_2	50% H_2, 50% N_2	[17]
			600	141	212			
			700	345	376			
		Ni-SDC//LSGM//SSC	500	8	22	50% NH_3, 50% N_2	50% H_2, 50% N_2	
			600	63	121			
			700	258	323			
		LSTNC-SDC//SDC//BSCF	650	120	218	100%	100%	[18]

[7] If it is reported 100%, the fuel was tested pure.

		700	190	305			
		750	266	393			
		850	361	479			
	LSTN-SDC//SDC//BSCF	800	161	338			
	LSTC-SDC//SDC//BSCF	800	98	222			
	Ni-SDC//SDC//BSCF	800	314	443			
	Fe-SDC/LSG/SSC	900	390				[19]
Anode	Ni-YSZ//YSZ//LSM-YSZ	650	86	94	100%	3%H_2O, 97%H_2	[20]
		750	299	305			
		850	526	530			
	Ni-SDC//SDC//BSCF	550	167	748	100%	100%	[21]
		600	434	1357			
		650	1190	1872			
	Ni-YSZ/Ni-SSZ//SSZ//LSM-SSZ	650	266	292	100%	100%	[22]
		700	451	462			
		750	654	741			
		800	1028	1204			
	Ni(97.5)Fe(2.5)-YSZ/Ni-SSZ//SSZ//LSM-SSZ	650	279	286	100%	100%	
		700	455	449			
		750	735	720			
		800	1150	1175			
	Ni-YSZ//YSZ//GDC-LSCF	600	97	135	66.7% NH_3, 1.7% H_2O, 31.6% N_2	60%H_2, 1% H_2O, 39% N_2	[23]
		700	329	361			
	Ni-SDC//SDC//SSC-SDC	500	65.1	82	100%	100%	[24]
		600	168.1	192			
		700	252.8	273			
	Ni-YSZ//SYO (0.1)-60YSZ	600	240	-	100%	100%	[25]
		800	1210	-	100%	100%	
	NiO-YSZ//PZO	800	1220	-	100%	100%	[26]
	YSZ	800 (1 atm)	1078	-	100%	100%	[27]
		850 (1 atm)	1174	-	100%	100%	
		800 (3 atm)	1148	-	100%	100%	
		850 (3 atm)	1202	-	100%	100%	
	Ni/YSZ/GDC-LSCF	760	300	-	0-100%	0-100%	[28]
	NI/SDC/BSCF	550	167	-	42.9% NH_3 1.4% H_2O 55.7% N_2	45% H_2 1% H_2O 54% N_2	[29]
		600	434	-	42.9% NH_3	45% H_2	

						1.4% H_2O 55.7% N_2	1% H_2O 54% N_2	
			650	1190	-	42.9% NH_3 1.4% H_2O 55.7% N_2	45% H_2 1% H_2O 54% N_2	
		BSCF	450	65	135	100%	100%	[30]
		BSCF	750	390	465	100%	100%	[30]
	-	Ni-CGO/BCGO/BSCFO-CGO	600	147	172	100%	100%	[31]
	-	Ni-CGO/BCGO/BSCFO-CGO	650	200	223	100%	100%	[31]
	-	BCG	700	25	28	100%	100%	[32]
	-	BCGP	700	35	37	100%	100%	[32]
Tubular	Electrolyte	Ag (filled with Fe)//YSZ//Ag	...	...	...	7.70%	10%	[33]
Tubular	Electrolyte	Ni-YSZ//YSZ//Ag	800	10	...	100%	100%	[14]
Tubular	Anode	Ni-YSZ//YSZ//LSM-YSZ	800	200	202	100%	3%H_2O, 97%H_2	[34]
Tubular	Anode	Ni-YSZ//YSZ//LSM-YSZ	700	38	44	100%	3%H_2O, 97%H_2	[35]
Tubular	Anode	Ni-YSZ//YSZ//LSM-YSZ	800	62	82	100%	3%H_2O, 97%H_3	[35]
Tubular	Anode	Ni-YSZ//YSZ//LSM-YSZ	900	88	118	100%	3%H_2O, 97%H_4	[35]

Table 8- Main experimental results of long term – durability tests

Cell type	Support	Temperature [°C]	Degradation [%]	Time (h)	Test - A/cm2	Ref.
Button	Anode	800	0	100	0.1	[25]
Tubular	-	750	1.32	50	0.2	[36]
Tubular	-	800	4	>100		[34]
	-	850	4	1500	1	[37]
	Anode	770	5	1000	0.38	[38]
	Anode	770	4	1000	0.38	[39]
	-	835	1.1		0.226	[40]
Button	-	600	0	50	0.36	[24]
	Electrolyte	900	1(1000h)	3000	0.39	[19]
	Anode	750	0.96 (1000h)	3000		

Amon – Ammonia to power

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