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List of Abbreviations

UNISA	Partner n°1
CEA	Partner n°2
SP	Partner n°3
IJS	Partner n°6
VTT	Partner n°7
EPFL	Partner n°10
MDPC	Monitoring, Diagnosis, Prognosis, and Control
LHV	Lower Heating Value
SRU	Single Repeat Unit
SS	Short Stack
SOFC	Solid Oxide Fuel Cell
SoH	State of Health
MTBF	Mean Time Between Failures
KPI	Key Performance Index
BoP	Balance of Plant
EIS	Electrochemical Impedance Spectroscopy
in	Inlet
out	Outlet
PH	Pre-Heater
FU	Fuel Utilisation
OU	Oxygen Utilisation
AU	Air Utilisation
TP	Top Plate
S/C	Steam to Carbon ratio in molar fractions
NTP	Normal Temperature and Pressure
THT	TetraHydroThiophene

List of Symbols

Quantity	Symbol	Unit
Hold time	t	h
Electrical current through the stack	i	A
Voltage of the stack	V	V
Gases flowrate	f	NI min^{-1}



Public Abstract

This deliverable summarizes the testing protocol of SOFC stacks and systems in normal and faulty conditions as agreed between the manufacturer (SP), testing partners (CEA, EPFL and VTT), and MDPC algorithms developers (UNISA and IJS). The aim of this testing campaign is threefold. Firstly, it would shed light on SOLIDpower stack and system behaviour under recurrent faulty operations. Secondly, it would provide validation means for MDPC algorithms development. Finally, it will give performance reference for actual MDPC tool validation campaign. For this purpose, the most relevant faults reported by SP from its on-field return of experience (WT3.1) have been selected, namely desulfurizer fault, water treatment system fault and fuel starvation. Moreover, the most conclusive monitoring techniques from the previous project INSIGHT have been retained.



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1. OBJECTIVE

This deliverable summarizes the testing protocol of a SOLIDpower stack and system in normal and faulty conditions. The objective of this work is to acquire clearer status of the stack and system's state of health with the least perturbation. The experimental results of these tests are planned to give validation means for the Monitoring, Diagnostic and Prognostic (MDP) algorithms developed in the frame of WP4, and to give performance reference for actual MDPC tool validation campaign carried out later in the project under WP7. These algorithms will be implemented on a controlling board in an objective to reduce the need for maintenance interventions of SP system, thus reducing the related costs, and improving its availability and life-time. This work is the follow-up and scale-up of INSIGHT project, where the focus was placed more on identifying the relevant metrics for monitoring Single Repeat units (SRU) and Short Stacks (SS) state of health.

The main features and specifications of the tested commercial products are listed in Table 1.

Table 1: Main features and specifications of the BlueGEN BG-15[®] system.

Manufacturer	SOLIDpower S.p.A.	
Commercial name	BlueGEN BG-15 [®]	
Application	μ-CHP	
Stack	Technology	SOFC
	Cells number	70
	Electroactive area	80 cm ²
	Nominal output voltage	55 Vdc
	Nominal output current	28 A
	Maximum stack voltage	74 Vdc
	Maximum stack current	34 A
System	Anode gas	Natural gas
	Cathode gas	Air
	Rated electric power	1.5 kW
	Rated thermal power	0.6 kW
	Fuel consumption	2.51 kW
	Maximum electric efficiency	55% (on LHV)
	Maximum thermal efficiency	25% (on LHV)
	Communication & Control	Remote
	Maintenance interval	12 months
	Lifetime	10 years
	Availability	97.5%

The document is organised into two main parts dedicated to stack testing planned at CEA and system testing at EPFL and VTT.



2. STACK TESTING

As natural gas pipeline is not currently available at CEA facilities, it was agreed to operate the stacks with methane that is partially reformed with steam generated by a boiler provided by CEA. In this case SP suggests a commercially available nickel-based steam reforming catalyst and to operate it at conditions where the methane is partially reformed. Methane conversion should be then measured at the stack inlet. For this purpose, sampling downstream the reformer, and performing gas composition analysis using a μ GC is required. It was also proposed by CEA in this frame to upscale its mass-flow controllers to handle higher steam flowrates (up to $f_{\text{H}_2\text{O}} \approx 10$ NL/min). It was also agreed to keep the electric power of the pre-reformer furnace constant throughout the testing campaign (and not compensate for the temperature fluctuations) in order to mimic the real system operating conditions.

The delivered stacks include a base plate that should be mounted on CEA's test bench. The base plate further provides connections for fuel and oxidant supply and exhaust, rods onto which the current leads can be connected, and voltage terminals to read out the stack voltage.

Considering the faulty operation, it is recommended to have a backup gas available to avoid detrimental failure of the stacks, e.g. forming gas while operating at OCV.

The SOLIDpower stack operation constraints are listed below. The following parameters should be monitored permanently, and faulty conditions should be ended if one of the conditions is not valid anymore:

- Stack exhaust temperature $< 780^\circ\text{C}$
- Stack voltage > 52.5 V (i.e. > 0.75 V/cell)
- Max 25mV decrease in steady-state operation on cell level
- Max 1.75V decrease in steady-state operation on stack level
- Stack current < 32 A
- Fuel utilisation $< 85\%$
- Oxidant utilization $< 30\%$
- Pressure drop in fuel line $< +25$ mbar.

Three full stack boxes will be shipped by SP to CEA in the frame of this project. Each stack box will be used to investigate each of the three faulty conditions which are relevant for the application. Three test programs were elaborated for each faulty condition and detailed in the following sections. If the stacks are still in an acceptable SoH at the end of each testing module, mixing multiple faults simultaneously could be considered.

2.1. Sulfur Poisoning



One of the main assets of SOFCs is their high fuel flexibility, which offers them the possibility to run on commercially available fossil fuels. However, such fuels may lead to anode poisoning caused by unfavourable reactions of catalytic anode materials with sulfur and/or carbon species present in readily available hydrocarbon fuel¹. In particular, sulfur poisoning has paid the most attention because of its immediate and severe detrimental effect on cell performance under various operating conditions. Sulfur can either be an impurity or an additive in several commercially available fuels and is present mainly in the form of hydrogen sulfide H₂S.

The aim of monitoring sulfur poisoning is to gain online insights about the desulfurizer state of health. This latest loses gradually its effectiveness with time, thus letting a proportion of hydrogen sulfide H₂S pass through the pre-reformer to the stack. It has been found by Matsuzaki et al.² that the minimum H₂S content after which the electrochemical degradation starts taking place depends strongly on the temperature. The threshold value was found at 0.05, 0.5, and 2 ppm (v) at 750 °C, 900 °C, and 1000 °C, respectively. It has been shown by Weber et al.³ via a DRT analysis that an H₂S-containing reformat affects the stack performance on three different levels, namely, electrochemistry in the anode functional layer, gas diffusion and water-gas shift in the anode substrate. Gas diffusion limitation is a consequence of water gas shift reaction being hindered by the Ni-surface catalyst deactivation by sulfur. In this case, CO and CO₂ are no longer involved in the reaction and act as an 'inert' components in the gas mixture. The gas diffusion is limited in this case to hydrogen and steam flows. Although, it should be noted that no physical obstruction of porosity is reported to be caused by sulfur poisoning.

If the methane content in the reformat is increased, the sulfur action is more pronounced and reforming into H₂ and CO is blocked. This situation, as reported by Weber et al., can also alter the thermal balance of the stack. For this reason, it is important to monitor the stack temperature for any eventual undesired fluctuations or gradients. A delay is systematically observed between H₂S supply and polarization resistance increase. This corresponds to the time needed for H₂S accumulation via chemisorption on Ni particles. Moreover, the operating temperature affects also the H₂S saturation and recovery time as reported by Matsuzaki et al. Indeed, saturation time is quicker at higher temperatures (3.3, 2.5, and 1.1 h at 750 °C, 900 °C, and 1000 °C, respectively), and recovery is faster as well at higher temperatures (100, 25, 1.1 h at 750 °C, 900 °C, and 1000 °C, respectively). The effect of H₂S content on saturation and recovery time is reported to be almost negligible by Matsuzaki et al., while a ratio of two was found for the H₂S content range tested by

¹ M. Gong, X. Liu, J. Trembly, C. Johnson, Sulfur-tolerant anode materials for solid oxide fuel cell application, *J. Power Sources*. 168 (2007) 289–298. <https://doi.org/10.1016/J.JPOWSOUR.2007.03.026>.

² Y. Matsuzaki, I. Yasuda, *Solid State Ionics*, 132 (2000) 261-269.

³ A. Weber, S. Dierickx, A. Kromp, E. Ivers-Tiffée, *Fuel Cells* 13 (2013) 487-493.

Papurello et al.⁴ (0.8-6.7 ppm). Finally, it was reported that prolonged exposure to H₂S (>24h) higher than 2 ppm(v) may lead to irreversible cell performance degradation.

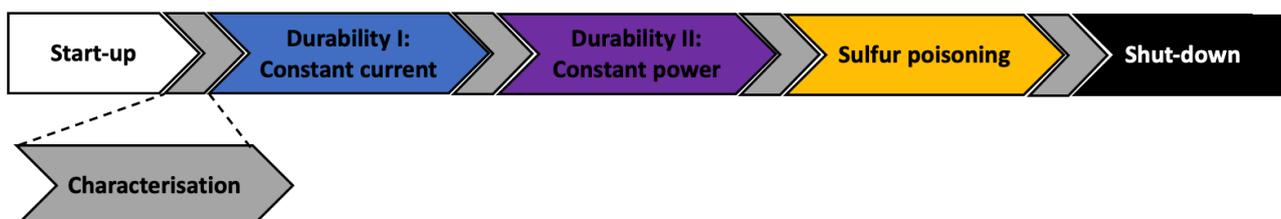


Figure 1: Overview of the test program for the investigation of fault I 'Sulfur poisoning'.

An adapted testing protocol is proposed, taking into account SOLIDpower stack specificities, and existing knowledge from literature and partners' experience. The initial sulfide content in the fuel was set to 0.125 ppm(v) (corresponding to 1.875 µg/min at NTP in nominal fuel feed conditions, c.f. Section 2.4) and then doubled in an iterative process, 0.25, 0.5, 1, up to 2 ppm(v). The exposure duration for the lowest sulfide contents of 0.125 ppm is relatively longer (48h) compared to the higher exposures (24h) in order to reach the initial surface coverage. A last high H₂S exposure of 4 ppm can be considered if the stack SoH is still in an acceptable level. Besides, the protocol includes two distinct testing sets, a first one where the injection is performed at the pre-reformer inlet and a second one at the stack inlet. This allows acquiring specific insights on how each component will react to a desulfurizer fault. A recovery phase is also foreseen between the two testing campaigns, which could be accelerated by increasing the pre-reformer and stack temperature. Gas outlet composition analysis should be performed for every tested component.

The exposure times indicated here are proposed only as a general indication and for guiding purposes. The actual exposure time should be adapted considering the different monitored metrics. It should be as long as required to reach the steady state and should be stopped prematurely if one of the previously stated stack constraints is breached.

During exposure, stack inlet and outlet will be analysed periodically (at least at the beginning and end of exposure sequence) to check composition. Gas samples will therefore be sampled and injected in a GC for analysis.

It is expected that the nickel will be specifically attacked in the Ni-based reforming catalyst. Therefore, in case the total nickel content in the catalyst bed is known, as well as the amount of sulfur that reaches the bed and the composition of the reformat produced by the reformer, it should be possible to quantify the sulfur poisoning effect. Indeed, the accumulated H₂S amount divided by the total accessible Ni-surface determine the onset of the degradation.

⁴ D. Papurello, A. Lanzini, S. Fiorilli, F. Smeacetto, R. Singh, M. Santarelli, Chemical Engineering Journal, 283 (2016) 1224-1233.



A sensitivity analysis over the most influencing parameters, besides the H₂S content, is also of capital interest. It is well established that temperature and current density are the most relevant parameters, affecting both electrode poisoning and recovery rate. It could be expected that lowering the temperature will help detecting sulfur poisoning earlier, and increasing it will stimulate recovery. Increasing current density could also accelerate both poisoning and recovery. These two parameters should be then swept in the range defined by SP ($700\text{ °C} < T < 800\text{ °C}$ and $0.2\text{ A/cm}^2 < i < 0.6\text{ A/cm}^2$) to investigate their effect on the stack, and their potential use for monitoring and recovery purposes. The detailed test program is given in Section 2.4 concerning the specification test modules.

At the end of the tests, the stack will be set back to the initial nominal conditions (without contaminants) and EIS measurements will be performed. A final *i-V* will be measured before switching to forming gas for the stack cooling (60 °C/h), or passing to the next testing module.

2.2. Carbon deposition

Besides sulfur poisoning, the commercially available hydrocarbon fuels are liable to induce a second degradation phenomenon commonly known as coking, or carbon deposition. Several experimental studies in simulated synthesis gas^{5,6,7,8,9} or methane^{10,11,12} perform thermodynamic analysis in order to identify the regions where carbon formation is favourable. A recent study by Kuhn and Kessler^{13,14}

⁵ M. Drewery, E. Kennedy, F. Alenazey, B. Dlugogorski, M. Stockenhuber, The effect of synthesis gas composition on the performance of Ni-based solid oxide fuel cells, *Chem. Eng. Res. Des.* 101 (2015) 22–26. <https://doi.org/10.1016/J.CHERD.2015.07.008>.

⁶ H. Miao, G. Liu, T. Chen, C. He, J. Peng, S. Ye, W.G. Wang, Behavior of anode-supported SOFCs under simulated syngases, *J. Solid State Electrochem.* 19 (2015) 639–646. <https://doi.org/10.1007/s10008-014-2640-7>.

⁷ Y. Zhang, Z. Yang, M. Wang, Understanding on the carbon deposition on the Nickel/Yttrium–Stabilized Zirconia anode caused by the CO containing fuels, *J. Power Sources.* 279 (2015) 759–765. <https://doi.org/10.1016/J.JPOWSOUR.2015.01.074>.

⁸ T. Chen, W.G. Wang, H. Miao, T. Li, C. Xu, Evaluation of carbon deposition behavior on the nickel/yttrium-stabilized zirconia anode-supported fuel cell fueled with simulated syngas, *J. Power Sources.* 196 (2011) 2461–2468. <https://doi.org/10.1016/J.JPOWSOUR.2010.11.095>.

⁹ V. Alzate-Restrepo, J.M. Hill, Carbon deposition on Ni/YSZ anodes exposed to CO/H₂ feeds, *J. Power Sources.* 195 (2010) 1344–1351. <https://doi.org/10.1016/J.JPOWSOUR.2009.09.014>.

¹⁰ P. Fan, X. Zhang, D. Hua, G. Li, Experimental Study of the Carbon Deposition from CH₄ onto the Ni/YSZ Anode of SOFCs, *Fuel Cells.* 16 (2016) 235–243. <https://doi.org/10.1002/fuce.201500038>.

¹¹ Y. Jiao, L. Zhang, W. An, W. Zhou, Y. Sha, Z. Shao, J. Bai, S.-D. Li, Controlled deposition and utilization of carbon on Ni-YSZ anodes of SOFCs operating on dry methane, *Energy.* 113 (2016) 432–443. <https://doi.org/10.1016/J.ENERGY.2016.07.063>.

¹² J. Millichamp, T.J. Mason, N.P. Brandon, R.J.C. Brown, R.C. Maher, G. Manos, T.P. Neville, D.J.L. Brett, A study of carbon deposition on solid oxide fuel cell anodes using electrochemical impedance spectroscopy in combination with a high temperature crystal microbalance, *J. Power Sources.* 235 (2013) 14–19.

¹³ J. Kuhn, O. Kesler, Carbon deposition thresholds on nickel-based solid oxide fuel cell anodes I. Fuel utilization, *J. Power Sources.* 277 (2015) 443–454. <https://doi.org/10.1016/J.JPOWSOUR.2014.07.085>.

¹⁴ J. Kuhn, O. Kesler, Carbon deposition thresholds on nickel-based solid oxide fuel cell anodes II. Steam:carbon ratio and current density, *J. Power Sources.* 277 (2015) 455–463. <https://doi.org/10.1016/J.JPOWSOUR.2014.07.084>.



investigated threshold for different operating parameters i.e. FU, S/C ratio, current density and temperature. Their thermodynamic threshold calculations were based on the assumption that carbon is deposited as solid graphite. They concluded that above 700°C thermodynamic data can be used to predict thresholds, while thresholds below 600°C strongly disagree. Similarly, He et al.¹⁵ and Lee et al.¹⁶ came to the conclusion that thermodynamic, physical and kinetic properties of graphite cannot solely explain the details for anode degradation. Different forms of deposited carbon are reported ranging from nanotubes and fibers to dissolved carbon¹⁷ and furthermore whisker-type structures¹⁸.

Yet, the major aspects determining coking are certainly (i) temperature¹⁹ and (ii) polarization^{20,21,22}.

The proposed test program includes the same start-up procedure and initial testing period as outlined in sulfur poisoning module (characterization + durability). After ‘the baseline’ test period with nominal S/C-ratio = 2.0, carbon deposition will be caused on purpose for verifying different fault detection methods. The carbon deposition will be introduced by stepwise decrease of the S/C ratio from 2.0 down to 1. Five steps are considered: 2.0 – 1.8 – 1.6 – 1.2 – 1.0 with 18 h hold time per step. However, it may be necessary to adjust the testing time if failure (i.e. fast voltage decrease/break down of cell voltage) is detected earlier.

In order to assess the influence of operating conditions on coking, and more importantly on its detectability, it was suggested to perform a sensitivity analysis over the most influencing parameters besides the S/C ratio, i.e. **current density** and **temperature**. For this purpose, the protocol proposed earlier should be performed on the nominal stack temperature of 750 °C and current density of 0.38 A/cm², and also within a range of values, 700 °C < T < 800 °C and 0.2 A/cm²

¹⁵ H. He, J.M. Hill, Carbon deposition on Ni/YSZ composites exposed to humidified methane, *Appl. Catal. A Gen.* 317 (2007) 284–292. <https://doi.org/10.1016/J.APCATA.2006.10.040>.

¹⁶ W.Y. Lee, J. Hanna, A.F. Ghoniem, On the Predictions of Carbon Deposition on the Nickel Anode of a SOFC and Its Impact on Open-Circuit Conditions, *J. Electrochem. Soc.* 160 (2013) F94–F105. <https://doi.org/10.1149/2.051302jes>.

¹⁷ H. He, J.M. Hill, Carbon deposition on Ni/YSZ composites exposed to humidified methane, *Appl. Catal. A Gen.* 317 (2007) 284–292. <https://doi.org/10.1016/J.APCATA.2006.10.040>.

¹⁸ A. Lanzini, P. Leone, C. Guerra, F. Smeacetto, N.P. Brandon, M. Santarelli, Durability of anode supported Solid Oxides Fuel Cells (SOFC) under direct dry-reforming of methane, *Chem. Eng. J.* 220 (2013) 254–263. <https://doi.org/10.1016/J.CEJ.2013.01.003>.

¹⁹ T. Chen, W.G. Wang, H. Miao, T. Li, C. Xu, Evaluation of carbon deposition behavior on the nickel/yttrium-stabilized zirconia anode-supported fuel cell fueled with simulated syngas, *J. Power Sources.* 196 (2011) 2461–2468. <https://doi.org/10.1016/J.JPOWSOUR.2010.11.095>.

²⁰ Y. Lin, Z. Zhan, J. Liu, S.A. Barnett, Direct operation of solid oxide fuel cells with methane fuel, *Solid State Ionics.* 176 (2005) 1827–1835. <https://doi.org/10.1016/J.SSI.2005.05.008>.

²¹ Y. Lin, Z. Zhan, S.A. Barnett, Improving the stability of direct-methane solid oxide fuel cells using anode barrier layers, *J. Power Sources.* 158 (2006) 1313–1316. <https://doi.org/10.1016/J.JPOWSOUR.2005.09.060>.

²² J. Liu, S.A. Barnett, Operation of anode-supported solid oxide fuel cells on methane and natural gas, *Solid State Ionics.* 158 (2003) 11–16. [https://doi.org/10.1016/S0167-2738\(02\)00769-5](https://doi.org/10.1016/S0167-2738(02)00769-5).

$i < 0.6 \text{ A/cm}^2$. It should be reminded here that the feeding gases temperatures should be adapted accordingly to limit the temperature gradient with the stack (maximum 20 °C).

As recovery strategy for carbon deposition the stack will be set back to nominal operating conditions for 24 h. Intermediate and regular characterizations after each durability module, as well as after each fault introduction will allow life-time assessment and evaluation of counter-acting methods for each particular failure. The shut-down procedure is identical to the previous program.

N.B.: With respect to Ni-based reforming catalyst, it mainly depends on the chemical composition of the catalyst substrate, where it is known that alkalis and earth alkalis typically have a carbon mitigating effect. In addition, a pre-reformer regeneration strategy could be to increase the temperature and going back to the nominal steam to Carbon ratio.

A schematic description for the proposed test program 'Carbon deposition' can be taken from Figure 2.

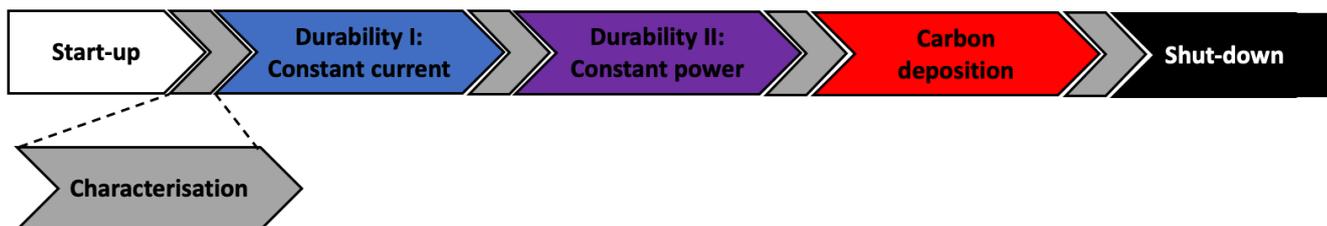


Figure 2: Overview of the test program for the investigation of fault II 'Carbon deposition'.

2.3. Fuel starvation

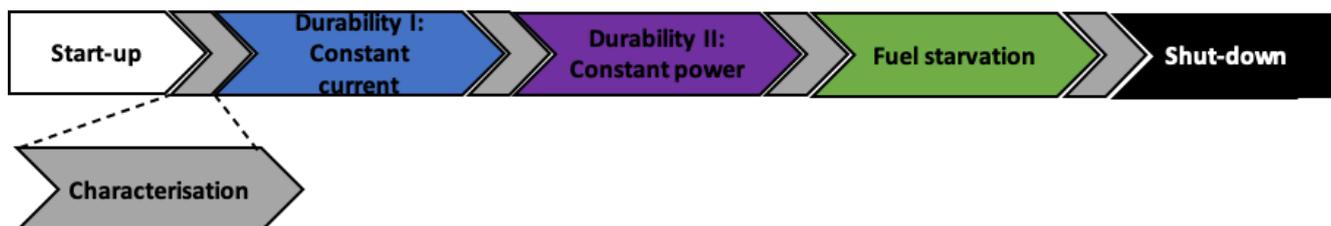


Figure 3: Overview of the test program for the investigation of fault III 'Fuel starvation'.

There are two possible ways to mimic fuel starvation; either by increasing the current density while maintaining the same fuel feed, or *vis-versa*, i.e. by lowering the fuel flow while maintaining the same current. The first method corresponds to a 'software bug', and is less statistically common from the on-field return of experience, while the second method relates to a 'hardware fault' whereby a physical feeding problem is encountered. It was agreed then to adopt the more common scenario, where a fuel feed is no longer ensured at the desired level. Practically, it corresponds to testing the stacks and systems at different reformate flow rates while keeping the S/C ratio constant.



The detailed proposed test program is illustrated in Figure 3. The test program begins with the start-up ($120^{\circ}\text{C h}^{-1}$) of the pre-reduced stack with a constant fuel feed of forming gas, and an initial characterization based on the guidelines given from the SOCTESQA-project (iV + EIS at OCV and 30.5 A). A durability module at constant power conditions is then performed in order to investigate stacks close to real operating conditions. These modules serve as 'long-term' baselines for further characterization and fault investigations.

Afterwards, the last of three relevant faults '*Fuel starvation*' will be introduced. The designed experiment targets to simulate the break-down of the fuel supply (hardware fault) under constant operating conditions of the full-stack. '*Fuel starvation*' will be caused by stepwise throttling of the fuel supply, with regular monitoring of each step (18 h per step are proposed for sufficient investigation time). Each step decreases the fuel supply by 5%, so that a characterization at 75% - 80% - 85% - 90% - 95% FU will be investigated. If stack operation constraints not fulfilled anymore, the fuel supply will be increased to nominal conditions (75% FU) and kept for 18 h. Then a characterization of the stack is performed for evaluating the degree of recovery after the fault. This protocol can be performed at a lower temperature of 700°C and a higher temperature of 800°C to assess its effect on fuel starvation and its detectability. Thereafter, we suggest performing an additional durability module in order to correlate degradation behaviour of a stack at 'fault free' conditions and 'faulty' conditions for life time prediction.

Detailed specifications for each module are outlined in the next section 'Specification test modules'.

2.4. Specification test modules:

Module	Details	Specifications	Monitoring	Time	Remarks
Stack Start-up	start-up	$f_{\text{Air,in}}$: 0 to 160 NI min^{-1} (Air) T_{stack} : RT to 300 °C (2 °C min^{-1}) $T_{\text{reactants}}$: RT to 300 °C (2 °C min^{-1}) Fuel composition: forming gas T_{stack} : 300 °C to 700 °C (2 °C min^{-1}) $T_{\text{reactants}}$: 300 °C to 700 °C (2 °C min^{-1}) <u>Warning</u> : Temperature difference between stack and reactants must not exceed 20 °C. Temperature stabilization => at least 30 min. $f_{\text{Air,in}}$: 160 to 190 NI min^{-1} (Air) $f_{\text{CH}_4,\text{in}}$: 1.5 to 4.95 NI min^{-1} (CH_4) ($\text{FU}_{\text{stack}} = 0.751$) $f_{\text{H}_2\text{O},\text{in}}$: 3.0 to 9.90 NI min^{-1} (H_2O) ($\text{S/C} = 2.0$)	$T_{\text{stack,inlet}}$ $T_{\text{stack,out}}$ i V_{stack}	≈7h	This procedure applies to a pre-reduced stack, otherwise reduction procedure should be followed first.
	initial characterisation	$f_{\text{H}_2,\text{in}}$: 20.06 NI min^{-1} $f_{\text{N}_2,\text{in}}$: 13.13 NI min^{-1} $f_{\text{Air,in}}$: 370 NI min^{-1} Load stack at 30.5 A (2 A min^{-1}) while adjusting the furnace temperature to reach a stack temperature of 715 °C. i -V curve acquisition: 30.5 A -> OCV -> 30.5 A (2 A. min^{-1}) <u>Optional</u> : EIS measurement Recommended parameters: DC current: $I = 0 \text{ A} / 30.5 \text{ A}$	$T_{\text{stack,in}}$ $T_{\text{stack,out}}$ i V <u>Optional</u> : Z' Z''	≈1h + (≈1.5h)	Initial electrochemical characterization to check the health of the stack. A comparison with the qualification sheets should be done to check that there were no problems related to the stack transport between partners.

Module	Details	Specifications	Monitoring	Time	Remarks
Stack Characterization: constant current	<i>iV</i>	<p>Recommended parameters:</p> <p><u>flows</u> $f_{\text{CH}_4,\text{in}}$: 4.95 NI min⁻¹ $f_{\text{H}_2\text{O},\text{in}}$: 9.90 NI min⁻¹ (S/C=2.0)</p> <p>$f_{\text{Air},\text{in}}$: 190 NI min⁻¹ (Air)</p> <p><u>current control</u> i: 0 to i_{max} (< 32 A) i is varied stepwise with a ramp of 2 A.min⁻¹ i_{max} is reached when V_{stack} is not any more above 52.5 V (i.e. i_{max} is the last current step before the voltage drops below 52.5V)</p> <p>hold time at i_{max} as short as possible</p>	$T_{\text{stack,in}}$ $T_{\text{stack,out}}$ i V	≈30min	initial <i>iV</i> -curve
	EIS	<p>Recommended parameters:</p> <p>DC current: $i = 0 \text{ A} / 30.5 \text{ A}$ (included due to power density evaluation)</p> <p>@ 30.5 A: 75.1% FU</p> <p>$f_{\text{H}_2\text{O},\text{in}}$: 9.90 NI min⁻¹ (S/C=2.0) $f_{\text{CH}_4,\text{in}}$: 4.95 NI min⁻¹</p> <p>$f_{\text{Air},\text{in}}$: 190 NI min⁻¹ (Air)</p> <p>Hold $t=10\text{-}30$ min (temperature stabilization before EIS) Frequency range: 100 kHz – 20 mHz (start from 100 kHz) Number of frequency points per decade: 7 (Note: 4 points are also acceptable at low frequency in order to shorten the measurement time)</p> <p>EIS should be performed in galvanostatic mode.</p> <p>Amplitude of AC current: $\bar{I} = 1 \text{ A}$ (Amplitude of AC voltage: $\bar{V} = 0.7\text{-}2.1 \text{ V}$) (NOTE: parameters can be adapted when appropriate in order to obtain for example impedance data of good quality) Set DC current stepwise to 0 after EIS</p>	Z' Z'' $ Z $ ϕ T_{stack} V_{stack}	7.5h	test specifications have been chosen based on the 'SOCTESQA'-project

Module	Details	Specifications	Monitoring	Time	Remarks
Stack Durability I: constant current	constant current operating conditions	<p>DC current = 30.5 A ; 75.1% FU</p> <p>$f_{H_2O,in}$: 9.90 NI min⁻¹ (S/C=2.0)</p> <p>$f_{CH_4,in}$: 4.95 NI min⁻¹</p> <p>$f_{Air,in}$: 190 NI min⁻¹</p> <p>Recommended impedance parameters at Frequency range: 100 kHz – 20 mHz (start from 100 kHz)</p> <p>Number of frequency points per decade: 7 (NOTE: parameters can be adapted when appropriate in order to obtain for example impedance data of good quality)</p> <p>Recommended parameters: DC current $i = 30.5$ A Frequency range: 1 Hz – 20 mHz (start from 1 Hz) Frequency amplitude: 5% and 10 % (1.5 and 3.0 A) Number of frequency points: 10</p>	<p>V_{stack} $T_{stack,in}$ $T_{stack,out}$ $(f_{neg,out})$ $(f_{pos,out})$</p> <p>optional: EIS monitoring each 48 h (4 IS for reference)</p> <p>alternate: PRBS monitoring each 6 h (4 measurements for reference). The frequency of performing PRBS and EIS should be adapted depending on the operating conditions.</p>	200h (or as long as needed to reach steady-state operation)	baseline for 'normal operating conditions' before fault detection



Module	Details	Specifications	Monitoring	Time	Remarks
Stack Durability II: constant power	constant power operating conditions	<p>Electrical power = 1.5 kW Set S/C = 2.0 and regulate $f_{CH_4,in}$ and $f_{H_2O,in}$ to 75.1% FU $f_{Air,in}$: keep air flow at 18.6% AU</p> <p>During operation regulate current and flow to keep 80% FU (and balance with N_2 to have equal net-flow)</p> <p>Recommended impedance parameters at Frequency range: 100 kHz – 20 mHz (start from 100 kHz) Number of frequency points per decade: 7 (NOTE: parameters can be adapted when appropriate in order to obtain for example impedance data of good quality)</p> <p>Recommended parameters: DC current $i = 30.5$ A Frequency range: 1 Hz – 20 mHz (start from 1 Hz) Frequency amplitude: 5% and 10 % (1.5 and 3.0 A) Number of frequency points: 10</p>	<p>V_{stack} $T_{stack,in}$ $T_{stack,out}$</p> <p>optional: EIS monitoring each 48 h (4 IS for reference)</p> <p>alternate: PRBS monitoring each 6 h (4 measurements for reference). The frequency of performing PRBS and EIS should be adapted depending on the operating conditions.</p>	200h (or as long as needed to reach steady-state operation)	<p>baseline for 'normal operating conditions' before fault detection</p> <p>N.B.: There is a common mismatch between the actual FU and the imposed one because of the fluctuations induced by the intrusive impedance spectra measurements.</p>

Module	Specifications	Monitoring	Hold time	remarks
Stack Fault I – Sulfur poisoning	$C(\text{H}_2\text{S}) = 0 \text{ ppm}$	EIS, PRBS and all available electric and thermodynamic data.	24h	H ₂ S-free fuel. A constant bias will be applied during the test (no setting to OCV). <u>Sensitivity analysis on T:</u> In the range of 700-750-800. Apply monitoring techniques for every temperature-step.
	$C(\text{H}_2\text{S}) = 0.125 \text{ ppm(v)}$ Corresponding to 2.5 µg/min at NTP. Considering an H ₂ S concentration of 1.363 g/L	EIS, PRBS	48h	Injecting hydrogen sulfide (H ₂ S) progressively. A relatively long exposure time (48h) is planned for this low H ₂ S concentration as it requires longer time to reach a plateau.
	$C(\text{H}_2\text{S}) = 0.25 \text{ ppm(v)}$	EIS, PRBS	24h	H ₂ S exposure doubled every step. Hold time 24h or up to reach a plateau.
	$C(\text{H}_2\text{S}) = 0.5 \text{ ppm(v)}$	EIS, PRBS	24h	
	$C(\text{H}_2\text{S}) = 1 \text{ ppm(v)}$	EIS, PRBS	24h	
	$C(\text{H}_2\text{S}) = 2 \text{ ppm(v)}$	EIS, PRBS	24h	
	Optional: $C(\text{H}_2\text{S}) = 4 \text{ ppm(v)}$ Applicable if the stack is at its end of lifespan, because irreversible damage may occur. Or as short as possible to avoid permanent damage.	EIS, PRBS	24h	
	go to step 1 conditions	EIS, PRBS	48h	H ₂ S-free fuel to investigate recovery possibility (expected recovery time dependant on temperature)

Module	Details	Specifications	Monitoring	Hold time	Remarks	
Stack Fault II – carbon deposition	S/C = 2.0	$f_{\text{Air},\text{in}}: 190 \text{ NI min}^{-1}$; $f_{\text{CH}_4,\text{in}}: 4.95 \text{ NI min}^{-1}$ $700 \text{ }^\circ\text{C} < T_{\text{stack}} < 800 \text{ }^\circ\text{C}$; $0.2 \text{ A/cm}^2 < i < 0.6 \text{ A/cm}^2$ Set current and change temperature in the selected range. Repeat for different currents.	$f_{\text{H}_2\text{O},\text{in}}: 9.90 \text{ NI min}^{-1}$	EIS, PRBS	18h	Step 0: Nominal conditions
	S/C = 1.8		$f_{\text{H}_2\text{O},\text{in}}: 8.91 \text{ NI min}^{-1}$	EIS, PRBS	18h	Step 1: gradually reduce steam flow.
	S/C = 1.6		$f_{\text{H}_2\text{O},\text{in}}: 7.92 \text{ NI min}^{-1}$	EIS, PRBS	18h	Step 2
	S/C = 1.4		$f_{\text{H}_2\text{O},\text{in}}: 6.93 \text{ NI min}^{-1}$	EIS, PRBS	18h	Step3
	S/C = 1.2		$f_{\text{H}_2\text{O},\text{in}}: 5.94 \text{ NI min}^{-1}$	EIS, PRBS	18h	Step 4
	S/C = 1.0		$f_{\text{H}_2\text{O},\text{in}}: 4.95 \text{ NI min}^{-1}$	EIS, PRBS	18h	Step 5
	recovery		go to step 0 conditions	EIS, PRBS	24h	recovery

Module	Details	Specifications	Monitoring	Hold time	Remarks	
Stack Fault III – fuel starvation	75% FU	DC current = 30.5 A; $f_{\text{Air},\text{in}}: 190 \text{ NI min}^{-1}$ Change temperature in the selected range.	$f_{\text{CH}_4,\text{in}}: 4.95 \text{ NI min}^{-1}$ Balance $f_{\text{H}_2\text{O},\text{in}}$ to keep S/C=2.0	EIS, PRBS	18h	step 1: 75% nominal condition
	80% FU		$f_{\text{CH}_4,\text{in}}: 4.65 \text{ NI min}^{-1}$ Balance $f_{\text{H}_2\text{O},\text{in}}$ to keep S/C=2.0	EIS, PRBS	18h	Step 2: 80% lower methane flow progressively.
	85% FU		$f_{\text{CH}_4,\text{in}}: 4.37 \text{ NI min}^{-1}$ Balance $f_{\text{H}_2\text{O},\text{in}}$ to keep S/C=2.0	EIS, PRBS	18h	step 3: 85%
	90% FU		$f_{\text{CH}_4,\text{in}}: 4.14 \text{ NI min}^{-1}$ Balance $f_{\text{H}_2\text{O},\text{in}}$ to keep S/C=2.0	EIS, PRBS	18h	step 4: 90%
	95% FU		$f_{\text{CH}_4,\text{in}}: 3.91 \text{ NI min}^{-1}$ (92.5% FU) Balance $f_{\text{H}_2\text{O},\text{in}}$ to keep S/C=2.0	EIS, PRBS	18h	step 5: 95%
	recovery		go to step 1 conditions		18h	recovery

Module	Details	Specifications	Monitoring	Hold time	Remarks
Stack Shut-down	Final characterization	<p>Load stack from actual current to 30.5 A (2 A.min⁻¹) while adjusting the furnace temperature to reach a stack exhaust temperature of 750 °C.</p> <p><i>i</i>-V curve acquisition: 30.5 A -> OCV -> 30.5 A (2 A min⁻¹)</p> <p><u>Optional</u>: EIS measurement Recommended parameters: DC current: $I = 0 \text{ A} / 28 \text{ A}$</p>	<p>V_{stack} T_{furnace} $T_{\text{stack,in}}$ $T_{\text{stack,out}}$ $T_{\text{reactants}}$</p> <p><u>Optional</u>: Z' Z''</p>	<p>≈1h + (≈1.5h)</p>	<p>The final characterization helps identifying eventual degradation.</p>
	shut-down protocol	<p>$f_{\text{Air,in}}$: actual value to 160 NI min⁻¹ (Air) $f_{\text{CH}_4,\text{in}}$: actual value to 1.0 NI min⁻¹ (CH₄) $f_{\text{H}_2\text{O},\text{in}}$: actual value to 2.475 NI min⁻¹ (H₂O) (S/C = 2.2)</p> <p>T_{stack}: actual value to 300 °C (2 °C min⁻¹) $T_{\text{reactants}}$: actual value to 300 °C (2 °C min⁻¹) <u>Warning</u>: Temperature difference between stack and reactants must not exceed 20 °C.</p> <p>Switch off cathode air flow and anode fuel flow: $f_{\text{Air, pos,in}}$: 160 NI min⁻¹ to 0 NI min⁻¹ (Air) $f_{\text{CH}_4,\text{neg,in}}$: 1.0 NI min⁻¹ to 0 NI min⁻¹ (CH₄)</p> <p>T_{stack}: 300 °C to RT (2 °C min⁻¹) $T_{\text{reactants}}$: 300 °C to RT (2 °C min⁻¹) <u>Warning</u>: Temperature difference between stack and reactants must not exceed 20 °C.</p>		<p>≈ 7h</p>	



3. SYSTEM TESTING

Two commercial systems, named BlueGEN BG-15, will be manufactured by SOLIDpower and delivered to EPFL and VTT, one each. Minor modifications are planned to be made for the systems to enable testing some faulty conditions as explained in the dedicated sections below. Nevertheless, the same components (stack, BoP and interface board) as the commercial product will be used. Moreover, SP agreed to calibrate the flow controllers provided in the system, and to measure the baseline performance prior to dispatch. The testing partners will also perform an initial performance characterization to check if there is any degradation related to product transportation and installation.

BlueGEN BG-15 system accessible data by testing partners:

- Process air flow (Burner and cathode) [NI/min]
- Fuel flow (Burner and Anode) [NI/min]
- Inlet Stack Temperature (cathode) [°C]
- Outlet Stack Temperature (cathode) [°C]
- Burner Temperature [°C]
- Reformer water flow [ml/min]
- Fuel utilization [%]
- Steam to Carbon [-]
- Power Output [W]
- Current Stack current [A]

BlueGEN BG-15 system controllable parameters by testing partners:

- Power setpoint (in power control mode)
- Current setpoint (in current control mode)
- Water flow rate setpoint
- Gas composition

BlueGEN BG-15 system requirements and constraints (more details available in the user manual):

- Water feed.
- Natural Gas feed at the pressure range recommended in the user manual.
- Internet access.
- Electricity supply.
- The fuel composition should be initially given to the BG-15 system via its integrated board, which will allow computing some internal parameters like FU.
- BG-15 can be both monitored and controlled online, without any safety issues.

For evaluation purposes, it is important to consider the Key Performance Indicators (KPIs) expected at the end of the project. Table 3 summarizes the target values compared with the current values.



The SP BlueGEN BG-15 typically has a maximum efficiency of 55% and is designed for a 10 years operation. Commonly, the system is operated until the efficiency is 50%. The lifetime could be extended of 25% if the target efficiency at End-of-Life (EOL) would be set to an average value of 35%. Moreover, at constant load the efficiency of SP BlueGEN BG-15 with RUBY tool is expected to increase of +1% (i.e., up to 56%). Thanks to the advanced control functions, it is foreseen an increase of +2% of the efficiency at part-load operation (51+2% @1000W; 43+1% @500W).

The KPIs presented in Table 2 will be collected during the 1-year validation tests by SP and will then be verified by comparing the collected values with those related to the conventional systems (i.e., not equipped with the MDPC tool).

Table 3: KPIs for SP μ -CHP; the values foreseen are set assuming the expected improvement performance in a 5 years scenario (2019-2023) built making use of the learning curve of the company, which is based on the historical data available for the previous μ -CHP

Parameter	Unit	BlueGEN BG-15	FCH 2 JU targets			BlueGEN BG-15 with RUBY tool
			2020	2024	2030	
Lifetime	years of operation	10	13	14	15	15*
Availability	% of the appliance	97.5	97	97	98	98.5
Durability stack	Hrs	40000	50000	60000	80000	60000
Reliability	MTBF (hrs)	25000	50000	75000	100000-	60000
El. Efficiency**	%LHV	55	35-60	37-63	39-65	56
Maint. costs	€ Ct/kWh	6	5	3.5	2.5	4

* RUBY will facilitate the enhancement of this target thanks to the advanced control of BOP.

** RUBY will improve the efficiency at part load (51+2% @1000W; 43+1% @500W).

As the system has automatic start-up and shut-down procedures, no protocol is needed in this case.

For the system testing in normal conditions, the protocol starts with 1000 h of stable operation under nominal operating point (1.5 kW), then at an intermediate power of 1kW during e.g. 200 h and finally at the minimum power of 0.5 kW during 200 h as well. This would help characterizing the efficiency figures defined within RUBY targets (cf. Table 4). Besides, it enables to extrapolate the system durability based on this initial stable operation.

A sensitivity analysis aiming at identifying the optimal electrochemical characterization settings is then required. Indeed, based on the impedance spectra acquired in the frame of INSIGHT project on SP's EnGen-2500 system, a noise was systematically encountered, especially at low frequency



perturbations²³. This noise could be encountered for the new BG-15 as well, which could make fault detection and identification a challenging task. The proposed solution is to acquire several impedance spectra and use their average. The goal would be then to identify the minimum number of EIS measurements required for an accurate fault detection.

Ideally, the impedance spectra (EIS & PRBS) measurement settings could be directly optimized to acquire a statistically representative spectrum. The parameters that could be optimized are as follow:

- The number of cycles of the periodic sinusoidal signals, especially at low frequencies around 10 mHz (this parameter could have the same effect as acquiring several spectra),
- The number of acquisition points per period,
- The amplitude of the signal (while keeping this parameter as low as possible to avoid being intrusive).

Once the system is tested in normal conditions and the sensitivity analyses performed, faults will be introduced deliberately to investigate the system response to each of them. As mentioned earlier, the most relevant faults identified by the manufacturer SP based on its on-field return of experience (c.f. D3.1.) are: desulfurizer fault, water flowmeter fault and fuel starvation. These faults will be introduced consecutively by raising order of severity.

3.1. Desulfurizer fault

In order to simulate a malfunctioning of the desulfurizer, SP will add an additional feed line for natural gas that blends in non-desulfurized natural gas with the natural gas that goes through the desulfurizer. This modification will be done for both systems intended for EPFL and VTT. It was agreed that EPFL and VTT will add a flow controller to one feed line to restrict the natural gas flow, and add a (passive) mass flow meter in the second line, in order to control the amount of S in the fuel.

In addition, SOLIDpower will make sure that there is a gas sampling point provided in both systems after both natural gas flows have come together, to allow EPFL and VTT to analyze the gas that is fed to the reformer.

However, a problem that arises is that NG contains other contaminants and odorants (besides H₂S) that are liable to intrude with the current study. The following Table 5 lists the odorants that are present in the NG pipelines in the two testing countries Switzerland (EPFL) and Finland (VTT).

²³ J.P. Ouweltjes, M. Gallo, P. Polverino, F. Galiano, S.N. Pofahl, P. Boškoski, G. Nusev, P. Caliandro, A. Leonardi, SOFC In-Field Test of a Tool for Advanced Monitoring, Diagnostics and Lifetime Prognostics, B0609, 14th European SOFC & SOE Forum 2020



Table 5: Odorants concentrations in the NG pipelines in the testing countries Switzerland (EPFL) and Finland (VTT)

Country	Odorant	Minimum odorant concentration*	Maximum odorant concentration	Typical odorant concentration
Switzerland ²⁴	THT	2.77 ppm (10 mg/m ³)	8.32 ppm (30 mg/m ³)	4.16-8.32 ppm (15-30 mg/m ³)
	S-Free Acrylate	2.24 ppm (8.8 mg/m ³)	-	3.05-3.56 (12-14 mg/m ³)
Finland	THT	-	5 ppm	3-4 ppm

*Concentration (ppm) = 24.45 x concentration (mg.m⁻³) / molecular weight. Assuming molecular weight (THT) = 88.171 g.mol⁻¹, and molecular weight (S-Free Acrylate) = 96.062 g.mol⁻¹

The composition of each odorants is given in the following Table 6:

Table 6: NG odorants composition

	Composition				%S
	THT	Ethyl Acrylate	Methyl Acrylate	2-Ethyl-3-Methylpyrazin	
Formula	C ₄ H ₈ S	C ₅ H ₈ O ₂	C ₄ H ₆ O ₂	C ₇ H ₁₀ N ₂	
Molecular weight (g mol ⁻¹)	88.2	100.1	86.1	122.2	
Sulfur-Free Acrylate	-	66%	32%	2%	0.0
THT	100%	-	-	-	36.4 %

Based on the odorant concentration table, the following table 7 summarizes the sulfur concentrations in the testing countries coming from the odorants.

Table 7: Sulfur concentrations in the NG pipelines coming from the odorants in the testing countries Switzerland (EPFL) and Finland (VTT)

Country	Minimum Sulfur concentration	Maximum Sulfur concentration	Typical Sulfur concentration
Switzerland	1 ppm	3 ppm	1.5-3 ppm
Finland	-	1.82 ppm	1.09-1.46 ppm

*Concentration (ppm) = 24.45 x concentration (mg.m⁻³) / molecular weight. Assuming molecular weight (S) = 32 g/mol

It should be noted here that even if the main source of sulfur in NG is the odorants, other sulfuric components, like hydrogen sulfide H₂S and Carbonyl sulfide COS, may be present as well. Moreover, the NG composition does not solely change between countries but also with time. This is partially explained by the fact that NG suppliers guarantee only a constant and stable calorific value by using different NG mixtures from different origins. As the exact sulfur content is not precisely known, it is

²⁴ Natural Gas odorization practices in Europe, Marcogaz technical association of the European natural gas industry.



highly recommended to check the actual sulfur content in NG, e.g., via a Gas Chromatograph (GC), before starting tests in faulty condition.

The proposed system testing protocol is similar to the one previously proposed for stack testing (c.f. Section 2.1). It starts with sulfur-free fuel composition, then sulfur is added in an increasing sequence. The exposure time for each step is set to 24h or up to reach a plateau. It is foreseen that the exposure time needed to reach a plateau may be longer than 24h for light sulfur exposure and shorter than 24h for higher sulfur concentrations. The first sulfur amount is set to 0.125 ppm(v), then this content is doubled in an iterative process, i.e. 0.25, 0.5, 1, up to 2 ppm(v). Just before the end of each step, the system is thoroughly characterized via polarization curve and EIS measurements at different current densities and temperatures. In order to assess the system's SoH after each exposition, it was suggested to go back to sulfur-free fuel feed condition after each step and quantify the recovery time and eventual permanent damage. Finally, if the system's SoH is still at an acceptable level, a higher sulfur amount of 4 ppm(v) was proposed to be tested for a short period of time depending on the observed voltage drop. At the end of this test campaign, the system should be set to nominal conditions for up to 200h, or as long as needed to reach a steady state. A detailed testing protocol table is circulated between the concerned partners.

3.2. Water flowmeter Fault

In order to simulate malfunctioning of the water flow meter, SOLIDpower's suggestion is to test the systems at different water flow setpoints while the natural gas flow is kept constant. However, it is expected that the controller won't allow critically low S/C ratios in carbon deposition tests in order to protect the system. It is therefore suggested to decrease gradually the water flow setpoint down to the minimum possible value where the system board will detect a fault and take over the control. The goal of this work is to check if the fault detection and isolation tools are able to detect and identify the nature and extend of the fault before the integrated BG-15 system board. This would reduce the need for maintenance interventions of BG-15 system, thus reducing the related costs, and improving its availability and life-time.

The proposed testing protocol is similar to the one proposed for stack testing (c.f. Section 2.2). It starts with nominal operating conditions (including nominal S/C ratio). Then, the water supply is decreased progressively in a way to reduce the S/C ratio by a 0.2 step, down to a value of 1.0. Every step is maintained for 24h or up to reach a plateau, and a recovery phase is planned between the steps to evaluate the system's SoH and recovery time, and quantify any potential permanent degradation. Just before the end of each step, the system is thoroughly characterized via polarization curve and EIS measurements at different current densities and temperatures. A detailed testing protocol table is circulated between the concerned partners.



3.3. Fuel supply Fault

In order to simulate fuel starvation, it was agreed to test the systems at different reformat flow rates while keeping the S/C ratio constant at the nominal value of 2.2.

However, it is expected that Bluegen's controller does not allow for FUs higher than 85 % to protect the system. In this case, the controller will probably lower the power output. Similarly to the previous fault, the goal of this work is to check if the fault detection and isolation tools are able to detect and identify the nature and extend of the fault before the integrated BG-15 system.

The suggested testing protocol is also inspired from the previous stack tests in faulty condition. It starts with nominal conditions including normal fuel supply. Afterwards, the fuel starvation is introduced by cutting progressively the fuel supply in a way to target higher fuel utilizations (75%-80%-85%-90%-95%). Similar to the previous faults, each step is held for 24h or more if needed in order to reach a plateau. Just before the end of each step, the system is thoroughly characterized via polarization curve and EIS measurements at different current densities and temperatures. Recovery phases are also planned between the faulty steps in order to evaluate the system's recovery time and quantify any induced permanent damage to the stack or the pre-reformer. The detailed system testing protocol is shared between the involved partners.

3.4. Detailed system testing protocol

Module	Specifications	Monitoring	Hold time	remarks
System Fault I – Sulfur poisoning	$C(\text{H}_2\text{S}) = 0 \text{ ppm}$	i-V, EIS, PRBS and all available electric and thermodynamic data.	24h	H ₂ S-free fuel. <u>Sensitivity analysis on T:</u> In the range of 700-750-800. Apply monitoring techniques for every temperature-step.
	$C(\text{H}_2\text{S}) = 0.125 \text{ ppm(v)}$ Corresponding to 2.5 µg/min at NTP. Considering an H ₂ S concentration of 1.363 g/L	i-V, EIS, PRBS	48h	Injecting hydrogen sulfide (H ₂ S) progressively. A relatively long exposure time (48h) is planned for this low H ₂ S concentration as it requires longer time to reach a plateau.
	$C(\text{H}_2\text{S}) = 0 \text{ ppm(v)}$	i-V, EIS, PRBS	48h	Recovery at nominal conditions to investigate the dynamics of stack and pre-reformer recovery.
	$C(\text{H}_2\text{S}) = 0.25 \text{ ppm(v)}$	i-V, EIS, PRBS	42h	H ₂ S exposure doubled every step. Hold time 24h or up to reach a plateau.
	$C(\text{H}_2\text{S}) = 0 \text{ ppm(v)}$	i-V, EIS, PRBS	42h	Recovery at nominal conditions.
	$C(\text{H}_2\text{S}) = 0.5 \text{ ppm(v)}$	i-V, EIS, PRBS	24h	
	$C(\text{H}_2\text{S}) = 0 \text{ ppm(v)}$	i-V, EIS, PRBS	24h	Recovery at nominal conditions.
	$C(\text{H}_2\text{S}) = 1 \text{ ppm(v)}$	i-V, EIS, PRBS	24h	
	$C(\text{H}_2\text{S}) = 0 \text{ ppm(v)}$	i-V, EIS, PRBS	24h	Recovery at nominal conditions.
	$C(\text{H}_2\text{S}) = 2 \text{ ppm(v)}$	i-V, EIS, PRBS	24h	
	$C(\text{H}_2\text{S}) = 0 \text{ ppm(v)}$	i-V, EIS, PRBS	24h	Recovery at nominal conditions.
	Optional: $C(\text{H}_2\text{S}) = 4 \text{ ppm(v)}$	i-V, EIS, PRBS	18h	Applicable if the stack is at its end of lifespan, because irreversible damage may occur. Or as short as possible to avoid permanent damage.
	go to H ₂ S-free fuel conditions	i-V, EIS, PRBS	200h	H ₂ S-free fuel to investigate recovery possibility (expected recovery time dependant on temperature)

Module	Details	Specifications	Monitoring	Hold time	
System Fault II – carbon deposition	Nominal S/C = 2.0	$f_{Air,in}: 190 \text{ NI min}^{-1}$; $f_{NG,in}: 4.6 \text{ NI min}^{-1}$ $700 \text{ }^\circ\text{C} < T_{stack} < 800 \text{ }^\circ\text{C}$; $i = 30.5 \text{ A}$	$f_{H2O,in}: 9.20 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	S/C = 1.8		$f_{H2O,in}: 8.28 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	Nominal S/C		$f_{H2O,in}: 9.20 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	S/C = 1.6		$f_{H2O,in}: 7.36 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	Nominal S/C		$f_{H2O,in}: 9.20 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	S/C = 1.4		$f_{H2O,in}: 6.44 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	Nominal S/C		$f_{H2O,in}: 9.20 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	S/C = 1.2		$f_{H2O,in}: 5.52 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	Nominal S/C		$f_{H2O,in}: 9.20 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	S/C = 1.0		$f_{H2O,in}: 4.6 \text{ NI min}^{-1}$	i-V, EIS, PRBS	24h
	Recovery at nominal S/C		$f_{H2O,in}: 9.20 \text{ NI min}^{-1}$	i-V, EIS, PRBS	200h

Module	Details	Specifications	Monitoring	Hold time	
System Fault III – Fuel Starvation	Nominal Fuel Supply (NFS)	$f_{Air,in}: 190 \text{ NI min}^{-1}$; $700 \text{ }^\circ\text{C} < T_{stack} < 800 \text{ }^\circ\text{C}$; $i = 30.5 \text{ A}$	$f_{CH4,in}: 4.6 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	95% of NFS		$f_{CH4,in}: 4.37 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	NFS		$f_{CH4,in}: 4.6 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	90% of NFS		$f_{CH4,in}: 4.14 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	NFS		$f_{CH4,in}: 4.6 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	85% of NFS		$f_{CH4,in}: 3.91 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	NFS		$f_{CH4,in}: 4.6 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	80% of NFS		$f_{CH4,in}: 3.68 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	24h
	recovery		$f_{CH4,in}: 4.6 \text{ NI min}^{-1}$ Balance $f_{H2O,in}$ to keep S/C=2.0	i-V, EIS, PRBS	200h