D3.1; Definition of protocols and criteria for qualification testing

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Executive summary

This deliverable defines relevant metrics, targets and corresponding protocols to qualify components and assemblies to be produced in GAMER. It integrates the metrics defined in the description of work in GAMER as well as additional ones, which will enable to qualify progress of manufacturing and integration of parts to form the single engineering unit (SEU) and further, the hydrogen reactor. Non-destructive protocols are selected. As these metrics and protocols are inherently dependent on the design of the SEU and reactor, some updates are foreseen in line with the designing phase and the process integration study carried out in WP1 and WP6.

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1 Introduction

1.1 GAMER project

The GAMER project aims at developing a novel cost-effective tubular Proton Ceramic Electrolyser (PCE) stack technology integrated in a steam electrolyser system to produce pure dry pressurized hydrogen. The electrolyser system will be thermally coupled to renewable or waste heat sources in industrial plants to achieve higher AC electric efficiency and efficient heat valorisation by the integrated processes. The project aims at establishing a high volume production of novel tubular proton conducting ceramic cells. The cells will be qualified for pressurized steam electrolysis operation at intermediate temperature (500-700°C). They will be bundled in innovative single engineering units (SEU) encased in tubular steel shells, a modular technology, amenable to various industrial scales. GAMER focuses on designing both system and balance of plant components with the support of advanced modelling and simulation work, flowsheets of integrated processes, combined with robust engineering routes for demonstrating efficient thermal and electrical integration in a 10 kW electrolyser system delivering pure hydrogen at minimum 30 bars outlet pressure.

Partners of GAMER are:

Partner (short name)	***Country***
SINTEF (SINTEF)	*Norway*
Coorstek Membrane Science AS (CMS)	*Norway*
CSIC, Instituto de Tecnología Química (CSIC)	*Spain*
Carbon Recycling International (CRI)	*Iceland*
University of Oslo (UiO)	*Norway*
MC2 Ingenieria y Sistemas SL (MC2)	*Spain*
Shell Global Solutions International B.V. (SGSI)	*Netherlands*

The consortium covers the full value chain of the hydrogen economy, from cell and SEU manufacturer (CMS), system integrators (MC2, CRI), through researchers (SINTEF, UiO, CSIC), to end users in refineries, oil and gas, chemical industry (CRI, SGSI, with advisory board members YARA and Air Liquide). All along the project, these experienced partners will pay particular attention to risk management (technical, economic, logistic, business) and ensure progress of the technology from TRL3 to TRL5. The overall consortium will perform strategic communication with relevant stakeholders in order to ensure strong exploitation of the project’s results.

1.2 Deliverable D3.1

This deliverable defines and describes protocols and criteria for qualification of primary components and assembled parts necessary for the construction of the hydrogen generator in GAMER. This entails verification of all mechanical, geometrical and functional parameters of all components. For the assembled parts, there will be two levels of qualification requirements:

i) "Pass Requirement" describes the performance/quality needed to reach GAMER target performance;
ii) "Acceptance Requirement" describes the minimal quality/performance defined by the partners, which allows at an initial stage of development further exploitation of the parts in the next steps of the validation process. This qualification is meant to avoid dead time and bottlenecks from possible lack of delivery of "passed" assemblies.

The quality control parameters and test protocols described herein serve as a starting point for the non-destructive validation of parts and assemblies through the definition of metrics of interest, which include those defined in GAMER, as well as other ones defined for qualifying the various stages of manufacturing and integration of components. Target values and test protocols will be updated based on the evolution of the SEU and hydrogen reactor design as well as on improved background knowledge of the complete system requirements as the project evolves.

In the following, we define the process flowsheets drafted to produce the U-bend of the SEU, the metrics associated with primary components and assembled parts, and finally, the protocols defined to quantify these metrics.

Notice that this work is based on component/assembly designs, which are gathered in a separate, confidential document (see Section 8.1).

2 Process flowsheet

In order to define the critical steps of manufacturing and parts assembly and associated metrics, the partners have drafted process flowsheets for the two types of SEU investigated in a first phase of GAMER. The main difference is based on the selection of the positrode:

- Type 1: the H_2O+O_2 electrode is based on BGLC/BZCY composite
- Type 2: the H_2O+O_2 electrode is based on LSM/BZCY composite

2.1 Cells with BGLC -BZCY based composite positrode

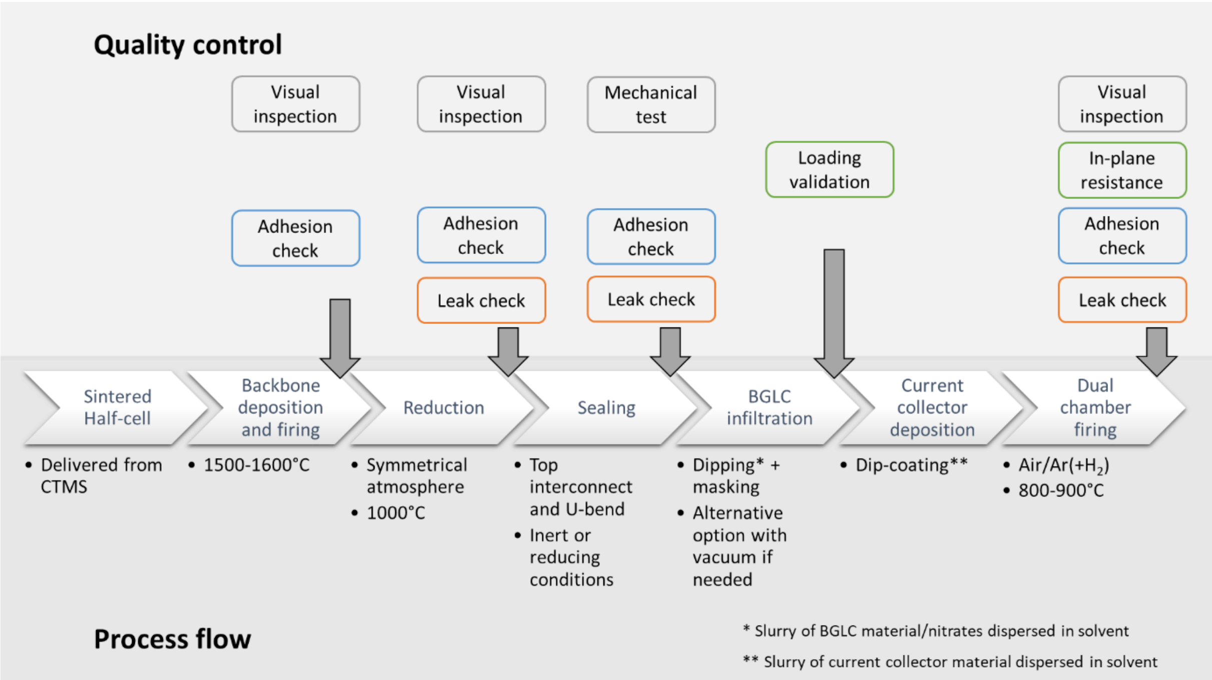


Figure 1: Process flowsheet of BGLC/BZCY based cells with corresponding quality control steps.

2.2 Cells with LSM -BZCY based composite positrode

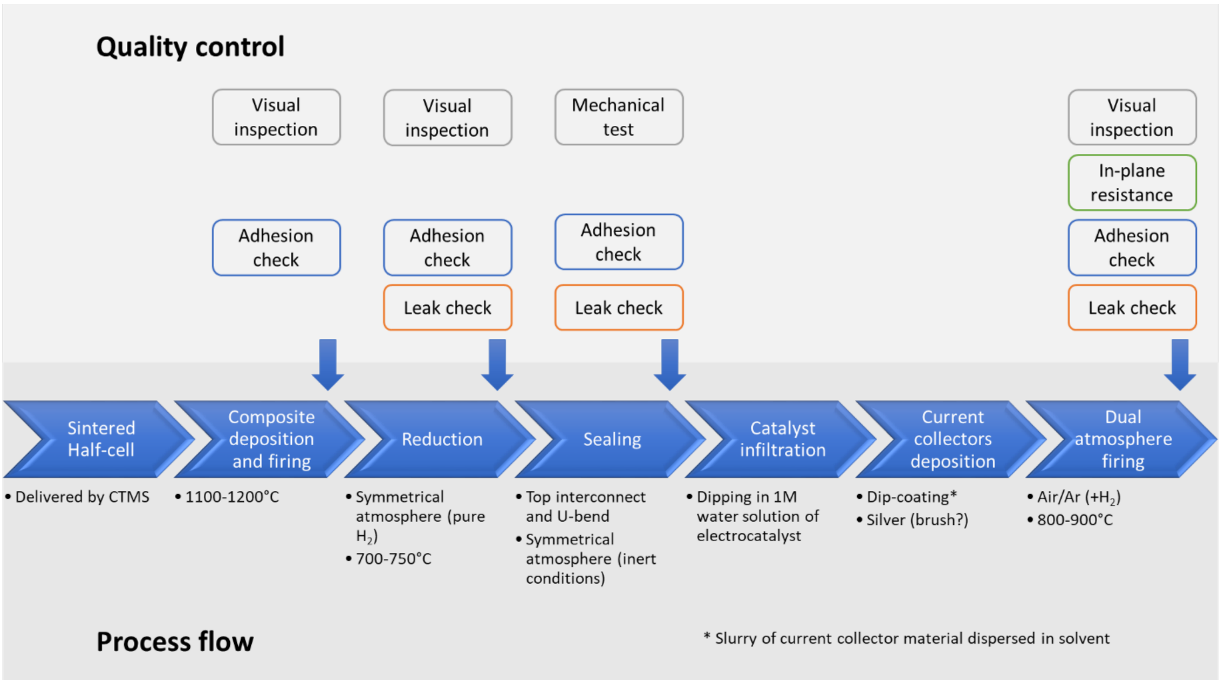


Figure 2: Process flowsheet of LSM/BZCY based cells with corresponding quality control steps.

3 Quality Control of Primary Components

Primary components refer to those designed by the partners and externally supplied, as well as the sintered half-cells with backbones made in the project.

3.1 Sintered half-cells

Description

Sintered tubular half-cell with Ni-BZCY based electrodes (serving as mechanical supports) and dense BZCY based membrane, optionally with porous BZCY based backbones.

QC metrics

Outer diameter	10.1 ± 0.3mm
Inner diameter	8.6 ± 0.2mm
Roundness	Within 3%
Length	Max 300 mm
Straightness	Pass through cylinder of 11 mm ID of equal length as half-cell
Leaks	No visible bubbles when submerged in isopropanol with internal overpressure of 1 bar
Adhesion:	No residue on tape following tape test (see ASTM D3359-17)

3.2 Outer seal

Description

Sintered glass ring of glass material 511 or 547, delivered according to specification

QC metrics

Outer diameter	13.2 ± 0.3 mm
Inner diameter	10.5 ± 0.2 mm
Thickness	2.2 ± 0.2 mm

3.3 Face seal

Description

Sintered glass ring of glass material 511 or 547, delivered according to specification.

QC metrics

Outer diameter	11.0 ± 0.2 mm
Inner diameter	7.3 ± 0.2 mm
Thickness	2.5 ± 0.2 mm (with centering ridge)

3.4 Two-tube and four-tube manifold boat

Description

Machined ZTA component (40 wt% ZrO_2), delivered according to specification (see Figure 1 in Annex I). Specifications subject to review.

3.5 Two-tube and four-tube manifold lid

Description

Machined ZTA component (40 wt% ZrO_2), delivered according to specification (see Figure 2 in Annex I). Specifications subject to review.

3.6 Two-tube and four-tube manifold (sealed)

Description

ZTA lid sealed to ZTA boat with GC 509 sealant

QC metrics

Seal integrity	Dye penetrant test

4 Quality Control of Assemblies

4.1 U- and W-bend assembly

Description

U- and W-bend consisting of two or four BCZY tubes with gradient connectors, sealed to two- or four-tube manifolds.

QC metrics

Mechanical strength	Acceptance: the bend can be easily manipulated
Internal resistance	Pass: Electrical contact across all interfaces
Membrane Resistance	Pass: Below 10 kΩ at room temperature at 1 kHz
Leaks	No visible bubbles when submerged in isopropanol with internal overpressure of 1 bar Pressure/vacuum decay test

* Individual materials validation and cell performance testing described in Annex I.

5 Quality Control of Reactor Components

Reactor design and dimensions depend on the outcome of the performance tests of outer electrode & current collector.
QC metrics to be determined when design and construction materials are finalized.

5.1 Reactor tube (pressure vessel)

Description

Assembly of reactor tube, lid and cap in steel. Specifications and pressure rating subject to final design of the reactor system, expected to be updated by M7.

QC metrics

Material	AISI 310 or equivalent
Outer Diameter	48 mm - This will depend on the vessel material and final SEU design (number of tubes and arrangement)
Wall thickness	3.5 mm
Length	400 mm (subject to review)
Reactor lid thickness	6 mm
Reactor cap thickness	6 mm

5.2 Electrical feedthrough

Description

Electrical feedthroughs for the SEU reactor tubes. Dimensions and specifications according to reactor design, expected to be updated by M7.

QC metrics

Length of alumina tube	
Diameter	
Wire material	
Wire diameter	
Yield	

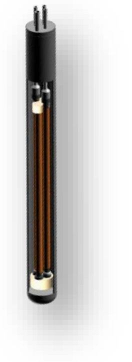
6 Quality Control of SEU and Hydrogen Reactor

QC metrics to be determined when design and construction materials are finalised.

6.1 U-bend in pressure vessel (SEU)

Description

SEU reactor with pressure vessel, electrical feedthroughs, several W bends.



QC metrics

Straightness of U-bend from lid
Clearance into reactor tube
Leaktightness of connection
Performance
Yield

7 Protocols for Quality Control

7.1 Geometrical measurements

Geometrical values of length, width and roundness are based on the average of three individual measurements every 6 cm using standard caliper. For roundness two measurements in perpendicular directions are carried out for each point.

7.2 Internal resistance

Measurement of internal resistance across outer current collector, gradient connector, interconnect, U/W-bend and internal current collector/electrode. Measured on assembled unit at room temperature using hand-held multimeter. Sufficient electrical percolation is achieved with resistance values below 1-2 Ω per U-bend (=60 cm cell + interconnectors), as considerable contact resistance is expected from the multimeter pins.

7.3 Sheet resistance – current collection layer

For validation of materials and processing procedures for current collectors, the sheet resistance will be measured on a separate single-cell setup. A four-point measurement setup is applied at both room temperature and at elevated temperatures (400°C to 700°C) in air (for positrode) and 5% H_2 (for negatrode).

7.4 Membrane resistance

For validation of the membrane resistance for U-bend and W-bend assemblies, cross plane impedance is measured from negatrode to positrode at 1 kHz using LCR meter. AC amplitude should not exceed 2 V during measurement.

7.5 Electrochemical performance

For validation of materials selection and processing procedure of complete single-cell assembly, the cell is reduced, sealed, capped and tested in a single-tube test reactor.

Test conditions:

Total pressure	3 barg
p_{H2O} (positrode)	1.5 barg
p_{O2} (positrode)	0.1 barg
p_{Ar} (positrode)	1.4 barg
p_{H2O} (negatrode)	0.1 barg
p_{H2} (negatrode)	0.5 barg
p_{Ar} (negatrode)	2.4 barg
Temperature	600°C
Voltage	1.3 V

8 Acknowledgements

This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking under grant agreement (number 779486). This Joint Undertaking receives support from the European Union's Horizon 2020 research and innovation program, Hydrogen Europe and Hydrogen Europe research.

9 Annex I

9.1 Schematics of parts: confidential

These schematics are available to GAMER project partners only. See document D3.1-Annex 8.1.

9.2 Materials validation of components and single cell assembly

Description

Complete assembly of cell with positrode and current collector, sealed and reduced inner electrode. Positrode comprising either a BZCY backbone infiltrated with BGLC or LSM-BZCY composite infiltrated with catalyst. Current collection layer comprising either Ag, oxide or composite mixture. Pass/Acceptance metrics are subject to revision pending the final design of the hydrogen generator.

QC metrics

Mechanical strength	Should be easy to handle
Outer sheet resistance	Acceptance: Contact across tube length when tested with multimeter at room temperature Pass: Below 20 mΩ across tube length at 600°C with 4pt measurement (subject to review)
Electrolyte resistance	Acceptance: 2 Ωcm^2 at 600°C and 1 atm H_2O Pass: 1.5 Ωcm^2 at 600°C and 1 atm H_2O
Electrochemical performance	Acceptance: 0.1 Acm^{-2} at 1.3 V Pass: 0.25 Acm^{-2} at 1.3 V
Leaks	No visible bubbles when submerged in isopropanol with internal overpressure of 1 bar